ABSTRACTS.

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Chemistry of Petroleum.

1. Catalysts for the Formation of Alcohols from Carbon Monoxide and Hydrogen. II., Synthesis of Methanol with Catalysts Composed of Copper and Zinc. P. K. Frolich, M. R. Fenske, P. S. Taylor and C. A. Southwich, Jr. *Ind. Eng. Chem.*, 1928, **20**, 1327—1330.

Previous work (Frolich, Fenske and Quiggle, *ibid.*, 694) has indicated that the decomposition of methyl alcohol in the presence of pure copper gives predominantly formaldehyde, that the addition of zinc oxide to the catalyst causes the formation of methyl formate, the yield of which is a maximum (58 per cent. of the alcohol decomposed) at *ca.* 88 mol. per cent. of copper, and that further addition of zinc oxide decreases the methyl formate yield with simultaneous increase in the yield of carbon monoxide. It was argued that those catalysts which yield carbon monoxide by the decomposition of methyl alcohol should promote the synthesis of the alcohol from carbon monoxide and hydrogen, whilst those catalysts should be inefficient for this synthesis which cause the production of methyl formate. This hypothesis is now tested and confirmed by experiments in which carbon monoxide, 26·5 per cent., and hydrogen 70·8 per cent. (0·6 per cent. oxygen, 2·1 per cent. nitrogen), are brought into contact at 204 atm. with catalysts prepared by the reduction of various mixtures of copper oxide and zinc oxide by means of methyl alcohol at 220° and atmospheric pressure. The sample of catalyst (3 c.c.), ground to 60 mesh, is supported by means of copper gauze in a brass reactor, the use of steel being objectionable because copper-zinc oxide mixtures are apparently very sensitive to poisoning by carbonyl. With each catalyst successive runs are made at 320, 350 and 370° and again at 320°. Molecular percentage composition of catalyst is plotted against percentage conversion of carbon monoxide to methyl alcohol. The general form of the curve is the same for 320, 350 and 370°, that for the second run at 320° showing a slight but measurable decrease in activity of the catalyst during the total cycle of operations lasting about 8 hrs. With increasing mol. per cent. of zinc oxide, the production of methyl alcohol increases, at first slowly, then rapidly at about 50 mol. per cent. of Zn O, a maximum being reached between 60 and 70 mol. per cent. of Zn O, the alcohol production falling off somewhat towards the point representing pure Zn O. The resemblance between these and the curve (obtained at 360 per cent. atmos. press.) connecting mol. composition of catalyst with per cent. of decomposed alcohol occurring as carbon monoxide, indicates that the more convenient decomposition method

(operated at atmospheric pressure) may be employed for investigating the activity of catalysts for the methyl alcohol synthesis. In both the synthetic and the decomposition experiments the results are obtained sufficiently far from equilibrium to be representative of the activity of the catalyst used. W. S. N.

2. Synthesis of Methane from Carbon Dioxide and Hydrogen. M. Randall and F. W. Gerard. *Ind. Eng. Chem.*, 1928, **20**, 1335—1340.

The equilibrium in the formation of methane and water vapour from carbon dioxide and hydrogen, and the reverse reaction, has been redetermined using reduced nickel, with and without ceria, as catalyst. The free energy of methane found from these experiments agrees with the value obtained by direct synthesis from hydrogen and graphite. W. S. N.

3. Synthesis of Higher Hydrocarbons from Water-Gas. II. D. F. Smith, C. O. Hawk and D. A. Reynolds. *Ind. Eng. Chem.*, 1928, **20**, 1341—1348; *cf. ibid.*, 462.

For details of results the original, which does not lend itself to abstraction, should be consulted. Water-gas, carefully freed from hydrogen sulphide, oxygen and carbon dioxide is passed over a copper-cobalt-manganese catalyst at (usually) 230—280°. A typical product obtained at 260° contains 21 per cent. of methane, 45 per cent. of enriching hydrocarbons ("gasol") and 34 per cent. of motor spirit. In general, most of the oxygen appears in the product as water, the ratio of which to carbon dioxide increases rapidly with decreasing temperature. At higher space velocities and temperatures relatively more olefines are produced, at low velocities and temperatures relatively more heavier hydrocarbons. In the course of a very complete theoretical discussion it is suggested that unsaturated hydrocarbons of low mol. wt. (the radicle CH_2 is mentioned) are primarily produced, which may then undergo hydrogenation or polymerisation or both. W. S. N.

4. The Pyrolysis of Methane. R. V. Wheeler and W. L. Wood. *Fuel*, 1928, VII., pp. 535–539.

This research was carried out in order to determine the optimum conditions for the formation of benzene from pure methane, since benzene was found to be an important product of the pyrolysis. The methods of experiment were similar to those of Fischer (*Brenn. Chem.*, 1928, 9, 309). Three types of experiments were made:—

I. *Static.*—Samples of methane were heated in bulbs of quartz in order to determine its decomposition point.

II. *Circulation.*—The course of the decomposition was followed from pressure changes that occurred when the gas was circulated in a closed system through a bulb of quartz at 900° and 950° C.

III. *Stream.*—Large volumes of methane were passed through tubes of different materials, at different rates and at different temperatures.

The apparatus used for the static experiments was similar to that used by Bone and Coward (*J. S. C. I.*, 1908, 93, 1197) in the thermal decomposition of hydrocarbons and later by Williams-Gardner (*Fuel*, 1925, 4, 430) for the pyrolysis of ethane.

From the results obtained it was concluded that:—

(*a*) Benzene is an important product of the pyrolysis of methane between 875° and 1100° C. Above 1000° C. carbon smoke accompanies the gaseous products of decomposition. The optimum temperature for the production of benzene appears to be about 1050° C., the best yield being 0·20 gals. per

1000 cub. ft. of methane passed through a horizontal reaction tube of quartz.

(*b*) Replacing the quartz tube by one of porcelain, or packing the tube with broken pieces of quartz produced no marked difference in the nature of the decomposition, the broken quartz surface having little or no effect on the formation of aromatic hydrocarbons by the pyrolysis of methane. Copper was found to exert but little catalytic effect on the decomposition.

(*c*) With an iron tube no condensable products of decomposition were obtained, the methane being decomposed into its elements.

(*d*) Dilution of the methane with nitrogen has little effect on the yield of aromatic hydrocarbons obtained at 1000° C., but dilution with hydrogen rapidly decreases the yield. H. E. T.

5. Methanol from Hydrogen and Carbon Monoxide. R. L. Brown and A. E. Galloway. *Fuel*, 1928, VII., 518–525.

A description of the apparatus used in the catalytic synthesis of methanol from hydrogen and carbon monoxide is first given, and then follow details of the preparation of the gas mixture and the three catalysts used during the experiments. The three catalysts are zinc oxide, basic zinc chromate, and normal zinc chromate.

Introductory experiments were carried out in order to obtain pressure-time curves for the three catalysts. The conditions employed were: closed circulating system; catalyst 250 c.c., system capacity (nett) 2000 c.c.; initial gas pressure 280 atmos. on the average, final, 100 atmos.; temperatures 300° C., 350° C. and 400° C.; make up gas none; rate of circulation 12·5 passes per hour. The resulting curves, together with corresponding ones showing the relation between pressure gradient (dP/dt) and pressure are given.

From these curves it was found that the rate of methanol production was low and practically constant at 310° C. for zinc oxide, being roughly insensitive to pressure changes, but responsive to temperature change. At 355° C. the activity has become such as to show dP/dt sensitive to pressure change, while at 400° C. a much greater activity is evident. In the case of normal zinc chromate, the curves show that the rate of methanol formation per volume of catalyst is materially faster at all three temperatures. For the basic chromate, the rate of methanol formation is also materially faster than for zinc oxide at the corresponding temperatures, and less than the normal salt at the upper range of temperature. The two chromates appear to reach, under the conditions employed, their maximum activity at 400° C. The curves also suggest that the "promoter" effect of the chromic oxide may be greater in the normal chromate at 350° C., and for the basic chromate nearer 300° C. For a strongly exothermic reaction like that of methanol, the question of temperature control becomes a very complicated one, and with a sizable amount of catalyst local overheating within the catalyst is encountered. This can be obviated by means of heat interchangers or other ingenious devices.

The liquid reaction products were examined as to weight, specific gravity, distillation characteristics and percentage of pure methanol. The percentage of methanol obtained was checked against an amount estimated from pressure drop and volume of the system, with proper allowance for temperature and other corrections.

The least amount of side reactions were found to take place at about 350° C. In the crude methanols formed, water was the chief impurity, while a few tenths of 1 per cent. of an oil were usually present. The principal gaseous product was methane containing traces of carbon dioxide, while with chromate catalysts there was a trace of ethane.

Constant Pressure Experiments.—A limited programme of comparative one hour tests at constant pressure of 200 atmospheres was outlined, the catalysts being tested at several temperatures. The tests at 400° C. have been completed.

Space velocities 3000, 75000 and 16,200 were used, and the methanol produced in one hour examined and weighed. The extent of drying of the gases was increased and the methanol produced regularly contained 97 $\pm$ 0·3 per cent. of pure product. The gas in the system at the outset contained 5 per cent. of total inerts, and the run was continued for from one to two hours according to the nature and activity of the catalyst employed. The resultant gas in all cases contained approximately 15 per cent. of inerts. The average effective pressure of gas was 180 atmospheres.

Life and Preparation of Catalysts.—The activity of the zinc oxide used in these experiments was found to decrease rapidly with use. In tests on the heat of reaction as to influence on the control of catalyst temperatures at various temperatures and space velocities, one batch of normal zinc chromate was employed for a period of 30 hours (17 hours at 300° C., 8 hours at 350° C., 3 hours at 400° C. and 2 hours at 425° C.). The decrease in activity of the catalyst over this period was 15 per cent., which is much less than that recorded for zinc oxide. The temperature of reduction of the catalysts during their preparation must be maintained as low as possible in order to obtain high activity. Observations on the relation of conditions of reduction to activity for basic zinc chromate are recorded.

Finally attempts have been made to correlate the experimental yields, with those calculated by thermodynamical methods, using Kelley's formula and subsequently the "Nernst Heat Theorem." H. E. T.

6. A Process for the Manufacture and Production of Hydrocarbons. I. G. Farbenind A.-G. E.P. 271,452, 1928.

Hydrocarbons or their oxygenated derivatives are produced by passing a mixture of hydrogen and an oxide of carbon—*e.g.*, water gas—over catalysts containing iron, cobalt or molybdenum produced from the corresponding metal carbonyls, the catalyst being preferably in the form of flakes. H. G. S.

7. Improvements in the Manufacture and Production of Benzene and its Homologues from Mixtures of Oxides of Carbon and Hydrogen. I. G. Farbenind A.-G. E.P. 297,179, 1928.

Benzene and its homologues are prepared by passing a mixture of oxides of carbon and hydrogen—*e.g.*, water gas—over a catalyst at temperatures exceeding 300° C., and under pressure and then heating the resulting gaseous olefines and gaseous homologues of methane under atmospheric or elevated pressure to 600° to 800° C. in presence or absence of catalysts. The catalyst for the first stage comprises iron and a noble metal or a difficulty reducible metal oxide, whilst that for the second stage must be free from materials which give rise to carbon. In the example quoted, water gas under 50 atmos. pressure is passed at a speed of 40 litres per hour per litre of catalyst over a catalyst prepared by melting in oxygen a mixture of iron powder and silver nitrate and reducing the mass at 450° to 500° C. with hydrogen. The catalyst is maintained at 370° C. during the passage of the water gas. The product, after freeing from CO_2, consists of 32 per cent. of Cn H_2n, 4 per cent. of CO, 5 per cent. of H_2, 25 per cent. of C_2H_6, 10 per cent. of CH_4 and 24 per cent. of N_2. This is passed through a copper tube filled with copper turnings free from iron at about 800° C. and a speed of 60 litres per hour per litre of catalyst, giving rise to a product which contains 80 per cent. of benzene.

H. G. S.

8. Manufacture and Production of Alcohols. J. Y. Johnson, Assr. to I. G. Farbenindustrie A.-G. E.P. 301,000. Nov. 19, 1928.

Alcohols, *e.g.*, a mixture of isopropyl, butyl and higher alcohols, are produced from the products of the destructive hydrogenation of mineral oil, coal, etc., their extraction, distillation and conversion products, etc., if necessary after a preliminary dehydrogenation, splitting or cracking treatment, by adding on water at the double bonds—*e.g.*, by adding sulphuric acid at ordinary or only slightly raised temperatures and then hydrolysing. *E.g.*, gas from the destructive hydrogenation of coal is compressed to about 40 atm. and the resulting liquid released to about 5 atm. The disengaged gas is brought into contact with moderately dilute sulphuric acid. The resulting sulphuric acid esters are hydrolysed by means of large amounts of water and the (isopropyl, butyl and higher) alcohols are distilled off.

W. S. N.

9. Manufacture and Production of Unsaturated Aliphatic Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenindustrie A.-G. E.P. 301,402. Nov. 27, 1928.

The conversion of aliphatic hydrocarbons (other than methane) or mixtures thereof by heat treatment in the gaseous or vapour state into unsaturated aliphatic hydrocarbons having the same number of carbon atoms per molecule is conducted in the presence of a catalyst consisting essentially of active charcoal, *e.g.*, wood charcoal prepared with the aid of zinc chloride, brown-coal coke activated with steam, active charcoal from lignite rich in humus, etc. The charcoal may be impregnated with metals or metallic compounds, or may be composed of (or prepared from) organic compounds of metals, *e.g.*, iron or zinc humate. Both mono- and diolefines may be prepared, it being often advisable when preparing a diolefine to operate in two stages. Other gases or vapours may be present with the hydrocarbon, and the operation may be conducted under atmospheric, reduced or increased, pressure. *E.g.*, the liquefied product from pentane treated at 450–500° with active charcoal (obtained by steaming brown-coal coke at 800–900°) contains, besides pentane, large quantities of amylene, with smaller amounts of other decomposition products.

W. S. N.

10. Catalytic Conversion of Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenindustrie A.-G. E.P. 301,969. Dec. 13, 1928.

The conversion of hydrocarbons into mixtures of carbon monoxide and hydrogen, by passing them with water vapour or carbon dioxide (with or without air and/or oxygen) over catalysts at elevated temperatures, is a highly endothermic process and, since it only occurs at high temperatures, t is difficult to introduce sufficient heat into the reacting materials. This difficulty is overcome by placing the contact substance (*e.g.*, nickel-alumina) in long chambers having a large heating surface, *e.g.* tubes of comparatively small cross-section (100 sq. cm. or less), the walls of the chambers which are externally heated being of heat-resistant, high heat-conductivity material, *e.g.*, highly alloyed steel, especially chromium-nickel steel, or high-grade nickel alloys such as chromium nickel. The process may then be conducted at temperatures below 1000°. With non-activated catalysts and very impure initial gases the flow of hydrocarbons and of heating medium is preferably counter-current; with contact material activated, *e.g.*, with alumina, and pure intial gases, the latter are best passed in the same direction as the external heating medium. For the production of mainly hydrogen and no carbon monoxide, catalysts which assist the conversion of carbon monoxide by means of steam (*e.g.*, those containing iron oxide and chromium oxide) may be mixed with the whole or part of the contact mass. If the initial

substances contain much organic sulphur compounds these must be removed by passage over a metallic mass (*e.g.*, copper) which absorbs them, or by the use of a contact substance (*e.g.*, metal oxides at 300°), which converts them to hydrogen sulphide, the latter being then removed in any suitable manner. The process (in which air or oxygen may be admitted with the hydrocarbons and steam or carbon dioxide) may be carried out in two stages (cf. E.P. 288,662). Examples are given showing the conversion of illuminating gas containing substantial quantities of methane and hydrogen into gases consisting largely of hydrogen, with smaller quantities of carbon oxides and only traces of methane. W. S. N.

11. Recovery of Volatile Organic Solvents. J. Y. Johnson, Assr. to I. G. Farbenindustrie A.-G. E.P. 302,297. Dec. 12, 1928.

The recovery of volatile organic solvents of many different kinds from gases containing the same is effected by washing with by-products of b.p. above 200° obtained in the catalytic hydrogenation of oxides of carbon to methanol or other oxygenated substances. This absorbent liquid does not take up water (hence the gases need not be dried), has low viscosity, low solidification point (below —60°, so that absorption may be carried out at a low temperature if desired), and low volatility, which makes a practically quantitative recovery possible by distillation, preferably in vacuo. W. S. N.

12. Separation of Gaseous or Low-boiling Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenindustrie A.-G. E.P. 302,203. Dec. 10, 1928.

The separation of unsaturated hydrocarbons of low b.p. (with or without other constituents), *e.g.*, cracked gases, may be facilitated by liquefaction under pressure, the resulting liquid being then fractionally distilled under elevated pressure either by gradually releasing the pressure or by raising the temperature or by a combination of both methods. *E.g.*, the vapour of cyclohexane is passed at 600° through a tube lined with quartz or firebrick and filled with calcium aluminate in cube form (cf. B.P. 297398), giving a gas containing about 28 parts by weight of ethylene, 14 of propylene, 4 of butylene and 40 of butadiene. The gas is liquefied by compression and expanded at room temperature to 15 atm., whereby ethylene alone is liberated. Heat is applied and expansion continued, propylene (98 per cent. pure) distilling at 25°/12 atm. The temperature is raised to 50° and expansion continued. Down to 6 atm. an intermediate fraction containing 50 per cent. butadiene and at 45°/5 atm. butadiene (98 per cent. pure) are obtained. Increasing the temperature an intermediate fraction, and finally at 52°/5 atm., practically pure butylene are obtained. A second similar example is also given. W. S. N.

Natural Gas.

13. The Effect of Temperature on Absorption Efficiency. A. J. L. Hutchinson. *Refiner*, 1928, 115.

From the work which has been carried out it is justifiable to assume that Raoult's law of solubilities holds. By this law, absorption varies inversely as the vapour pressure, and since vapour pressure varies directly as temperature, absorption must vary inversely as the temperature. This inverse ratio, however, is not a simple linear function.

By definition, the absorption efficiency is not the same at any temperature for two different constituents. In an absorber, the oil and gas approach

equilibrium at the bottom of the absorber and it is the conditions which exist at this point that have a large bearing on the efficiency. If an absorber extracts 60 per cent. of the butane with an oil circulation of 70 gallons per minute with the temperature of the oil at the bottom of the absorber at 100°, the same extraction efficiency for butane can be obtained with a temperature of 80° and oil circulation of 51 gallons per minute.

Under these conditions the absorption efficiency for propane will not remain constant but will decrease with the drop in temperature and lowering of the oil rate. With a circulation as above of 70 gallons of oil per minute in the absorber and a termperature of 100°, the absorption efficiency for propane would be 18·3 per cent., while at a circulation of 51 gallons per minute and a temperature of 80° F. it would be 12·9 per cent.

This appears to be in direct contradiction to general practice. It is the general opinion that in order to make a more stable product in the plant the oil temperature and oil circulation should be raised. However, the reverse is true. Both the oil temperature and oil rate should be lowered, taking care that the right ratio is maintained between temperature and rate. This point in the theory has been carefully checked in plant operations and found to be correct.

The vapour pressure of the constituents left in the oil from the still are higher, the higher the oil temperature. As absorption is a vapour pressure phenomena and depends on the lowering of vapour pressure due to solution, the higher vapour pressure of the constituents in the lean oil at higher temperatures will retard absorption. This, then, is another important reason for cooling the oil to as low a temperature as possible.

In practically all plants, an increase in oil circulation will raise the lean oil temperature to the absorbers. An increase in oil rate will usually lower the temperature rise through the absorber. Whether an actual gain in absorption efficiency for the useful constituents has been realised will depend entirely on the ratio of temperature increase to oil rate increase. S. W. C.

14. Ignition of Mixtures of Air with Natural Gas and with Methane by Induction Coil Sparks. E. G. Meiter. *Ind. Eng. Chem.*, 1928, **20**, 1353–1354.

The ease of ignition of methane and of Pittsburgh natural gas (88·9 per cent. CH_4, 7·4 per cent. C_2H_6, 2·4 per cent. C_3H_8, 0·8 per cent. C_4H_{10}, 0·1 per cent. higher hydrocarbons, 0·4 per cent. nitrogen) are compared by measuring the primary current necessary to give a secondary discharge (from a 15 cm. coil with motor-driven mechanical break) which will ignite known mixtures of each of the gases with air in the same glass ignition vessel (capacity about 38 c.c.), using bluntly pointed platinum electrodes 1 mm. apart. For each gas the primary current necessary for ignition is plotted against the percentage of gas in the air-gas mixture. The curves are very similar. Between 7·5 per cent. and 9·25 per cent. of methane the primary current is practically constant, rising sharply outside these limits. For natural gas the region of maximum ignition is 7·3 per cent. to 8·6 per cent., the primary current again rising sharply above and below these points. In qualitative agreement with the results of Wheeler (Trans., 1924, **125**, 1860) the current for the most easily ignitible air-natural gas mixtures is somewhat less than for the most easily ignitible air-methane mixtures. W. S. N.

15. Specific Gravity of Liquified Gas. G. G. Oberfell, T. W. Legatski, and F. E. Fisher. *Oil and Gas J.*, **27**, 33, 148.

The apparatus consists of a steel bomb, 3 inches in diameter and 12 inches long, fitted with two eduction pipes $\frac{1}{4}$ of an inch in diameter and each extend-

ing to within $\frac{1}{8}$ of an inch of the end of the bomb. Each pipe is fitted with a needle valve. The bomb is also equipped with an accurate pressure gauge reading to 500 lbs. to the square inch. Detailed specifications of connection, water bath, thermometers, balance and weights are also given. All threaded connections are made up permanently with litharge and glycerine to ensure the whole apparatus being leak-proof. The tare weight of the bomb full of air is first determined, then its water weight capacity at 60° F. The bomb is completely filled with distilled water by alternately evacuating it through one eduction pipe and then admitting water through the other, the bomb being maintained in an inverted position. The bomb is then placed in an ice bath for at least one hour, the water inlet pipe still being connected and the valve open, the evacuating valve being shut. At the end of this time the bomb is disconnected, all valves being closed, and placed in an accurately-controlled water bath maintained at 60° F. Any excess pressure over 100 lbs. per square inch is released by cracking one of the valves, and the bomb is kept in the bath until equilibrium is obtained. It is then removed, dried and weighed, the weight, less the tare, giving its water weight capacity at 60° F.

When dry and ready for use the bomb is purged with liquified gas until it contains nothing but vapour. The discharge pipe is closed and the bomb allowed to fill completely until the bomb gauge registers the pressure of the source of supply. All valves are now closed and the bomb disconnected. It is placed in the water bath maintained at 60° F., and a pressure approximately 100 lbs. to the square inch higher than the vapour pressure of the product at this temperature is maintained. Excess liquid is removed by cracking a valve as required. When equilibrium is attained the bomb is removed, momentarily chilled, dried and weighed. This weight less the tare weight is divided by the water weight capacity at 60° F. to give the specific gravity of the liquid at 60° F. Duplicate determinations should agree within 0·0008.

Obtaining equilibrium at 60° F. with the bomb full of liquified gas needs careful attention to prevent bursting the apparatus, but the writers state that a pressure relief valve is being developed to overcome this difficulty.

W. L.

See also Abstracts Nos. 43 and 52.

Motor Spirit.

16. Economical Recovery of Valuable Products from "Spent" Doctor and Caustic Solutions. F. J. Mechlin. *Ind. Eng. Chem.*, 1928, **20**, 1352–1353.

Spent doctor solution, *d* 1·19, containing 12·5 per cent. caustic soda, 0·3 per cent. of sodium sulphate, tarry materials of the type ROH, lead as plumbite ion, suspended lead sulphide and organic lead compounds (total PbO, 0·75 per cent.) and spent soda solution, *d* 1·06, containing 3·82 per cent. of caustic soda, 0·52 per cent. of sodium sulphate, and dissolved tarry matter, are evaporated and air-blown in an iron vessel until *d* reaches, respectively, 1·47 and 1·40, a final temperature of 132° in the liquid being necessary. Lead sulphide is gradually oxidised to lead sulphate, hence sodium sulphate and sodium plumbite are formed, the former separating from the concentrated liquid on settling. Colloidal organic material gradually agglomerates as an upper layer on the heavier caustic-plumbite solution. When the concentration reaches 35° Bé. a soapy layer separates, *d* 35–38° Bé., containing caustic

soda, sodium carbonate, sodium alcoholates and lead residues. This material is completely miscible with water and is somewhat soluble in gasoline. If it is not removed, on further concentration it is converted to a tar. The recovered caustic soda solution contains 23·0, 33·2 per cent. of NaOH (a recovery of 87·8, 94·7 per cent.) and traces of Na_2SO_4. The recovered plumbite solution contains 36·1 per cent. NaOH, 0·35 per cent. Na_2SO_4, 2·1 per cent. PbO, representing a recovery of 87·3 per cent. NaOH and 84·7 per cent. PbO.
W. S. N.

17. Process of Purifying Hydrocarbons. J. C. Black and W. H. Low, Assrs. to Pan-American Petroleum Co. U.S.P. 1,696,377. Dec. 25, 1928.

Cracked distillates are treated with cadmium hydroxide in aqueous alkaline solution, whereby cadmium sulphide and cadmium mercaptides are formed. The spent liquor is acidified, and the cadmium in the aqueous acid solution is recovered by adding excess of alkali. A plant is described for carrying out these processes continuously. W. S. N.

18. Process of Treating Liquids. L. Caldwell, Assr. to Celite Co. U.S.P. 1,691,266. Nov. 13, 1928.

The invention provides a process of de-sulphurising and de-colourising petroleum oils by mixing the oil, in the presence of water, with a pulverulent treating agent containing available chlorine. The treating material is obtained by the chlorination of the product of reaction of a mixture of diatomaceous earth and lime, such mixture containing free alkaline hydroxide prior to its treatment with chlorine. S. W. C.

19. Fractional Condensation of Mixed Vapours. L. Weisberg, Assr. to Barrett Co. U.S.P. 1,676,232. July 3, 1928.

The process provides for fractional condensation of mixed vapours, and consists of introducing the vapours into the lower-most portion of a fractionating device, condensing the total vapours issuing from the uppermost section, separating a portion of this condensate and returning the remainder to the uppermost section. The total condensate is withdrawn from the lower portion of each section in every section except the one in the lowest position, a portion of the condensate is separated and the remainder returned to the section next below. The ratio of the quantity of liquid so removed to the total of the reflux liquid should be maintained substantially constant irrespective of variations in the rate of flow of the total reflux liquid or the rate of flow of the ascending vapours. S. W. C.

See also Abstracts Nos. 4, 7, 43, 44, 49, and 81.

Kerosine.

20. Purification of Petroleum Hydrocarbons. A. G. Bloxam, Assr. to Allgemeine Gesellschaft für Chemische Industrie m.b.H. E.P. 301,955. Dec. 10, 1928.

Petroleum hydrocarbon distillates (illuminating oils) are purified (*i.e.*, the sulphur content is reduced and the odour improved) by extraction with liquid sulphur dioxide, the insoluble portion being freed from adhering sulphurous acid and treated with a dilute alkaline solution of lead oxide, with or without the addition of sulphur. *E.g.*, distillates from (1) Upton Co. Texas, (2) Smackover, (3) Hutchinson, (4) Sour Lake, having b.p. range,

194–256, 206–269, 207–260 and 195–250° and sulphur content 0·59, 0·21, 1·13 and 0·10 per cent. respectively, are treated with 85 per cent. (by volume) of liquid sulphur dioxide, and the insoluble portions, freed from sulphurous acid, are shaken with about 5 per cent. (by volume) of a solution of 3 per cent. lead oxide in dilute soda; the resulting oils are satisfactory in odour and have S content 0·12, 0·05 per cent., traces and 0·02 per cent. respectively.
W. S. N.

21. Improvements in and Relating to the Refining or Fractionating of Mineral Oil and Mineral Oil Products. Actien-Gesellschaft für Kohlensäure-Industrie and E. B. Auerbach. E.P. 277,946. Oct. 4, 1928.

The different degrees of solubility of the various hydrocarbons and their homologues in liquid carbon-dioxide are utilised as a means of refining mineral oils and mineral oil products. The oil is treated with about five times its weight of liquid carbon-dioxide in a closed vessel at atmospheric temperature. The oil-carbon-dioxide solution is separated from the residual oil, the carbon-dioxide gasified and reliquified ready for further use. The inventors claim that the disadvantages of existing refining methods are overcome, for, the process being carried out at ordinary temperatures, chemical decomposition, resinification and other causes of normal losses are minimised, also that the final products are free from all traces of the gaseous refining agent W. L.

22. Improvements in and Relating to the Refining or Fractionating of Oils. Actien-Gesellschaft für Kohlensäure-Industrie and E. B. Auerbach. E. P. 285,064. Oct. 25, 1928.

This is a modification of Patent No. 277,946. It is claimed that the solubility in liquid carbon dioxide can be used to separate mineral oils from fatty oils, fats and other hydrocarbon oils and chemically or physically similar products.

It is also claimed that fatty oils, fats, terpenes, tar oils, resin oils, ketone oils, essential oils and the like may be refined by treatment alone or separated from mixtures of phenol oils, fatty acids, colouring matter, odour-producing constituents and other similar compounds.

It would appear that the solubility of the various oils in liquid carbon dioxide depends not only on their chemical composition, but, with approximately the same composition, on the molecular magnitude.

Examples are given dealing with the separation of petroleum and rape-seed oil, the refining of crude rape-seed oil, crude amber oil and of the residue from the manufacture of turpentine, and with the fractionation of crude coal tar. W. L.

23. Fractionating Hydrocarbon. W. K. Lewis and A. A. Wells, Assrs. to Standard Oil Development Co. U.S.P. 1,672,849. June 5, 1928.

The invention provides for the separation of kerosine hydrocarbons from a gas oil distillate, which comprises vaporzing the distillate and bubbling the vapours through progressively cooler zones (one or more towers) countercurrently into washing contact with condensate produced by the cooling of the vapours further along in the course, while maintaining an operating pressure of 30 mm. of mercury. S. W. C.

24. Refining Oil. J. R. Neller and G. M. Vance, Assrs. to the Texas Co. U.S.P. 1,672,621. June 5, 1928.

The invention provides for a process of refining and stabilising hydrocarbon oils. It comprises treating the oil with sulphuric acid and separating

the resultant sludge from the oil. The latter is neutralised with an alkal reagent and then treated with a relatively small quantity of alcoholic alkal (less than 1 per cent. by volume) in proportion sufficient to remove potentiai colour-forming constituents from the oil. S. W. C.

See also Abstracts Nos. 16, 18, and 19.

Lubricating Oil and Wax.

25. Mechanical Filter-Aids in Refining. A. Eisenbast and F. L. Horine, Celite Products Co. *Oil and Gas J.*, **27**, 140.

Diatomaceous silica filter-aids can be used with advantage for a number of purposes in the handling of petroleum products.

The ordinary filter-aids (filter-cel) contains approximately 80 per cent. of SiO_2, about 4 per cent. of combined water and 5 per cent. of moisture. Two other grades are available which are free from moisture and are known as standard super-cel and hyflo super-cel. These bodies are without chemical action and have no bleaching properties. Their primary function is to build porous films against retaining mediums which are more efficient for filters than the mediums alone. Under identical conditions the rates of flow of water or oil through such porous films are in the proportions of one for filter-cel, two and one half for standard super-cel and five for hyflo super-cel.

In many filtering operations it has been found advantageous to pre-coat the filter cloth with a layer of the pure filter-aid, about 15 lbs. of which are necessary to form a layer of $\frac{1}{8}$ of an inch thickness over 100 square feet of filtering surface. The pre-coat is applied by pumping a suspension of the filter-aid in a clear liquid through the filter press.

For the removal of fine suspended matter or turbidity a small amount of the filter-aid is added to the oil to be filtered. From one to three times the weight of the solid to be removed or from 0·1 to 2 per cent. of the weight of the oil are the usual proportions employed. The mixture is thoroughly agitated and pumped to a filter press. The process enables impurities which would ordinarily pass through the cloth or would slime its surface and restrict the rate of flow to be removed readily. The method has been applied with success for breaking crude oil emulsions, precautions being taken to prevent the emulsion reforming after the filtration is complete.

Hyflo super-cel is used to facilitate the removal of amorphous wax or wax which is difficult to remove by ordinary filtration methods. In the production of a low pour test bright stock from a waxy cylinder stock, the wax-containing oil is diluted with naphtha. The naphtha solution is slowly chilled and a suitable amount of super-cel hyflo, depending on the amount and character of the wax, is added to the chilled stock with agitation. The mixture is then filtered cold. The filter-aid can be recovered from the wax cake and used again.

Filter-aids can be used with advantage in the filtration operation which follows the contact decolorisation of oils by finely divided adsorbent clays. When a filter press with metal cloth is used, the operation can be greatly facilitated by pre-coating the cloth with a suspension of the hyflo filter-aid in some filtered oil. Approximately 5 lbs. of filter-aid should be used per 100 square feet of filtering area. The main batch of oil containing the clay is then pumped through the filter without allowing the pressure to fall between the pre-coating and main operations. It is stated that the addition of 5 or 10 per cent. of filter-aid to suspensions of oils and decolorising clays before filtration, serves to form a more open cake, which retains less oil and does not restrict the rate of flow to the same extent as a cake of clay alone. The use of

filter-cel is also recommended for the removal of acid sludge from acid-treated oils where bleaching is not necessary. R. W. L. C.

26. COLD SETTLING. L. C. Jamieson. *Refiner*, 1928, 92.

Theoretically a tall narrow tank should be the best for cold settling as it concentrates the petrolatum bottoms in a narrower circle and hence more of the settled solution, which the author calls "top mix," can be skimmed off. On the other hand, a flat broad tank theoretically should settle out quicker, as the petrolatum on its way down through the solution would not have so far to travel. Actually a 30 × 25 tank of capacity 3000 barrels is the best "settler." Others of 1000 barrel capacity, size 20 × 20 do not compare either in chilling time or settling time per gallon received with the larger tank.

The commonest arrangement of chilling coils is a simple pipe coil with return bands at either end in one plane near the top of the tank. Others use coils in two planes, a few feet apart, or even in three planes, at top, centre and bottom of the tank. The author found that the fastest time could be made with coils in two planes, the top one within 2 or 3 feet of the top level of the solution and the second a few feet under the first one. Vertical coils are said to have the advantage that petrolatum does not freeze so readily on the coils themselves and thus retard the chilling effect of the brine in the coils. Another successful method is to chill the solution in a wax chiller as used in the press rooms until the temperature reaches 30–40°, then to fill the ordinary settler with its brine coils direct from the wax chiller, and carry on the chilling in the usual way.

To be successful in settling bright stock, the solution should be pre-heated to at least 90° to eliminate the moisture. The best method of doing this is to have two steam pumps, one for filtered solution and one for straight benzine, equipped with Mason governors. These pump into the same line, are passed through a cylinder with many baffle plates, the solution thus being completely mixed. From here the solution passes to the heat exchanger which was designed to heat 350 barrels of solution an hour to 100° and uses the exhaust steam from the pumps. From the heat exchanger the solution goes direct to the settler leading through the bottom draw-off line. The Mason regulators are set to give the required gravity and the entrance of the exhaust steam into the heat exchanger is handled through a valve and the temperature reading taken at the outlet of the heat exchanger. It has been found that within certain limits the gravity of the solution mix is not important except that if too much benzine is used, the percentage ot top mix received from the settler is too low. A solution of 44 gravity is aimed at, using 26·5 gravity steam-refined filtered stock and 56/8 gravity benzine around 400 end point. Roughly the precentage is 60 benzine to 40 filtered stock. The top mix can be skimmed from the settler either by the installation of permanent manifolds on the side of the tank, 6–8 inches apart, or by the use of swing lines.

The author has made a study of many conditions affecting cold settling and has arrived at the following conclusions: "Shock chilling" can probably be traced to bad mixing. Outside temperature has no effect upon the settling of an oil in a settling tank after the chilling has taken place. A solution of 43 gravity will settle out better than one of 46 gravity or higher, all other conditions being equal, and with due regard to percentage of top mix received. The introduction of bright stock washings into a settler solution to be chilled down apparently has no effect on the settling. A sudden drop over five points in the relative humidity seems to make a settler drop out in some 6–18 hours thereafter. This brings up the question of "why not make the required humidity inside the settling tank"?—a problem on which the author is now engaged. S. W. C.

27. Filter House Practice. J. P. Northrop. *Refiner.* 1928, 90.

There are many methods in operation in soaking and running filters, ranging from filtering under pressure up to 20 lbs. per sq. inch down to pumping the oil up through the bed of clay. Most of these give good results in the particular plants in which they have been perfected. Pressures up to 20 lbs. for filtering lubricating oils of a viscous nature are justifiable if for no other reason than the saving of time that can be accomplished both in soaking and running: by maintaining a back pressure on the bottom of the filter, channelling will be prevented with its loss of production.

The author is personally in favour of filtering all oils in solutions with as high as 50–60 per cent. naphtha. Much greater speed can be obtained in running time as well as draining, less naphtha consumed to thoroughly cleanse the clay and yield almost double that obtained on straight 600 stock, enabling the recycling period to be shortened by 24–48 hours per filter.

Due consideration should be given to the contact decolourising of lubricating oils in at least all new installations of any size. This method having been so perfected at the present time, its cost will compare very favourably with the percolation type of filter, even though no attempt is made to restore the powdered clay. Operations are more fully under control, and claims are also being made in restoring the fine clays to approximately the original efficiency by specially designed kilns which should make this of particular interest to all who are concerned with the cost of decolourising lubricating oils. S. W. C.

28. Separating from Fluid Hydrocarbons other Hydrocarbons which Precipitate at Low Temperature. N. O. Backlund, Assr. to Bergedorfer Eisenwerk Aktiengesellschaft. U.S.P. 1,676,069. July 3, 1928.

The invention provides for a process of separating from fluid hydrocarbons, other hydrocarbons which precipitate at low temperature and comprises the addition to the mixture of a liquid having a higher specific gravity than that of the heaviest of the other hydrocarbons (*e.g.*, trichlorethylene). S. W. C.

29. Process of Purifying Mineral Oils. M. L. Chappell, G. J. Ziser and E. L. Moyer, Assrs. to Standard Oil Co. U.S.P. 1,672,304. June 5, 1928.

The invention provides a method of purifying mineral oils after acid treatment, and consists in neutralising a viscous stock from an asphaltic base crude with an alcoholic caustic soda solution containing over 90 per cent. by volume of alcohol. The emulsifying constituents are removed by agitating the oil with an alcoholic solution containing over 90 per cent. of alcohol, and then separating the two liquids by decantation after stratification has taken place. S. W. C.

30. Process of Producing High-Viscous Lubricating Oil and High-Grade Asphalt from Petroleum. A. E. Pew, Junr., Assr. to Sun Oil Co. U.S.P. 1,675,462. July 3, 1928.

The invention describes a process of separating the heaviest and less heavy constituents of heavy residual mineral oil, and comprises the treatment of a shallow stream of oil to a low pressure (less than 1 in. mercury) and to a temperature above the vapourising point of the less heavy constituents, and simultaneously passing through the stream, a relatively light hydrocarbon thereby agitating the flowing liquid. This relieves surface tension and by entrainment effects maximum vapourisation of the less heavy con-

stituents. The heating is produced by flowing into heat exchange relation with the oil stream, mercury vapour at such temperature and pressure as to effect, by heat exchange and condensation of mercury vapour, the vapourisation of the less heavy constituents of the agitated and turbulent flowing stream. S. W. C.

31. Improvements in the Manufacture and Production of Viscous Oils. I. G. Farbenindustrie A.-G. E.P. 299,086. Oct. 22, 1928.

The invention provides a process for the production of viscous oils, which consists in passing gases rich in lower olefines into hydrocarbon mixtures, such as tar oils or mineral oils, which are liquid at ordinary temperatures, the mixture being treated at 120° C. in the presence of a catalyst of the Friedel-Crafts type, such as aluminium chloride. S. W. C.

32. Improvements in the Purification of Montan Wax. I. G. Farbenindustrie A.-G. E.P. 299,133. Oct. 25, 1928.

The invention provides a process for the manufacture and production of purified Montan wax, and consists in subjecting Montan wax in aqueous solution or suspension to the anodic action of an electric current in the presence of electrolytes and oxygen transferers and, if desired, of an organic solvent. For example, a lead vessel is charged with 40 per cent. sulphuric acid containing from 10–100 grams of chromium sulphate per litre. The lead vessel serves as the anode and is connected to the positive pole of a source of electric current. The cathode is made of any convenient metal which resists the action of the electrolytes and is preferably so constructed that it may be used as a stirrer. After the solution in the vessel has been warmed to about 90° C., the crude Montan wax is energetically stirred-in and the current switched on. The tension is from 3–5 volts, and the current density up to 500 amperes per square metre. The temperature is slowly raised to above 100° C., and the current density is slightly lowered towards the end of the bleaching process. When the Montan wax has been sufficiently decolourised, the current is switched off and the stirrer stopped. The purified Montan wax then floats on the surface of the liquid, and can be run off while hot or taken off when cold. S. W. C.

33. Process of Improving the Properties of Montan Wax. W. Pungs and M. Sahrstorfer, Assrs. to I. G. Farbenindustrie A.-G. U.S.P. 1,690,876. Nov. 6, 1928.

The invention provides for a process of improving the properties of Montan wax, and consists in boiling the wax with a concentrated aqueous solution of chromic acid in the presence of glacial acetic acid and sulphuric acid, the ingredients being used in the proportions by weight of 50 parts of Montan wax to 100 parts of glacial acetic acid to which have been added 1 to 4 parts of concentrated sulphuric acid and then 50 parts of chromic acid. The whole is boiled and stirred for some time, and then poured into hot water, when the cake of bleached wax is removed after cooling. S. W. C.

34. Improvements in Oxidising Paraffins, Waxes and the Like. J. Y. Johnson and I. G. Farbenindustrie A.-G. E.P. 298,704. Oct. 18, 1928.

This invention claims that the oxidation of paraffins, waxes, etc., by air in the presence of an inorganic catalyst, such as metals, metallic oxides or salts, including metallic salts of organic acids, may be considerably accelerated by associating organic nitrogenous bases with the above catalysts. Aniline, pyridine, quinoline, butylamine, toluidine and cyclohexylamine are men-

tioned as suitable nitrogenous bases. In the example given, paraffin wax oxidised by air in the presence of 1 per cent. of manganese soap at 170° C. gives a saponification value of 169.5 after 4 hours. If the 1 per cent. of manganese soap be associated with 0.1 per cent. of hexamethylenetetramine the saponification value after 4 hours amounts to 217. W. L.

35. Treatment of Mineral Oils. T. G. Delbridge and H. F. Dure, Assrs. to The Atlantic Refining Co. U.S.P. 1,690,772. Nov. 6, 1928.

The process describes a method of decolourising unrefined hydrocarbon oil, and comprises acid treating the oil whilst blowing with air. Water is added to settle the sludge formed, this treatment being followed by air blowing the acid treated unneutralised oil while at elevated temperature in order to condition it for clay treatment. Subsequently, finely divided clay is added to the unneutralised oil and intimately mixed at temperatures between 150° and 210° F. S. W. C.

See also Abstracts Nos. 21, 22, 24, and 78.

Special Products.

36. A Process for the Production of Aqueous Emulsions of Bitumen. Chemische Fabrik Buckau. E.P. 298,842. Oct. 18, 1928.

In this invention small amounts of acids and oxy-acids of boron, in alkaline solution, are employed as emulsion carriers. It is claimed that small amounts of oxy-acid of boron employed alone are superior to the emulsion carriers hitherto used, and that the best emulsions are obtained when the amounts of carrier used lie between 0·03 and 2·0 per cent.

Emulsions of this type display high stability, high concentration of bitumen, and a good and rapid setting capacity.

58 parts of bitumen heated to 120° C. is poured, with vigorous stirring, into a solution of 0·4 parts of boric acid and 0·5 parts of caustic soda in 41·1 parts of water heated to 100° C. W. L.

37. Improvements in or Relating to Bituminous Emulsions and to Their Use in the Coating of Substances with Bitumen. Colas Products, Ltd., W. S. Whiting and A. G. Terry. E.P. 301,544. Dec. 3, 1928.

This invention provides a method of applying bitumen to mineral aggregates by mixing the latter with an aqueous bituminous emulsion in the presence of an alkali salt.

The alkali salt must be a strong electrolyte, preferably sodium chloride or sulphate. (Ammonium salts are excluded.) Any aqueous bituminous emulsion, which is substantially stable in bulk, may be used. The alkali salt in the proportion of 0·5 to 2·0 per cent. on the weight of emulsion for sodium chloride or an equivalent proportion for other salts may be added in the form of solid or in aqueous solution. Also the alkali salt may be added to the aggregate prior to the incorporation of the emulsion or the emulsion may be mixed with the aggregate and the salt then incorporated.

If it is desired to retain the water liberated in coating aggregate with bitumen, a filler such as soft limestone may be added in the proportion of 4 per cent. or less on the weight of the aggregate.

The filler is added immediately after the water is liberated, and the resulting product remains in a workable condition for some time. In addition to mineral aggregates, cork, sawdust, clinker, asbestos and the like may be coated with bitumen by the method. W. L.

38. Improvements in or Relating to Bituminous Emulsions. J. A. Montgomerie. E.P. 300,415. Nov. 15, 1928.

This is a further development of E.P. 226,032, and deals with the production of an aqueous bituminous emulsion by mixing molten bitumen with a hot dilute aqueous solution of an oxidising alkali.

Molten asphalt is poured into a hot solution of sodium peroxide containing 0·5 per cent. of sodium peroxide.

The temperature employed is about 100° C. and the mixture is stirred continuously during the addition of the asphalt. It is believed that the liberated oxygen has an effect on the asphalt analogous to that obtained by preliminary air-blowing. The invention includes the employment of the oxidising alkali in admixture with other reagents. Stabilising agents such as sodium laurate may be added to the emulsion. W. L.

39. Improvements in the Production of Bituminous Emulsions. J. A. Montgomerie. E.P. 300,821. Nov. 22, 1928.

In this invention, a further development of E.P. 226,032, molten asphalt is poured, with continuous stirring into a hot dilute aqueous solution of the cresylate of one or more of the alkali metals with or without the presence of other reagents.

Both ingredients of the emulsion are kept at about 100° C. while mixing, and the cresylate is proportioned so as to give 0·5 per cent. NaOH reckoned on the bitumen.

To the emulsion obtained, stabilising agents such as sodium laurate may be added. W. L.

40. Improvements in the Production of Bituminous Emulsions. J. A. Montgomerie. E.P. 300,414. Nov. 15, 1928.

This invention, a further development of E.P. 226,032, claims that by employing normal sodium phosphate (Na_3PO_4), practically spontaneous emulsification of Mexican asphalt is effected.

Molten bitumen, of the type artificially prepared from petroleum, is poured into a hot solution of a tertiary alkali phosphate with continuous stirring. Both liquids are kept at about 100° C. The amount of normal sodium phosphate used is 0·5 per cent. reckoned on the bitumen.

The solution of phosphate and bitumen may be in equal proportions or otherwise.

The invention includes the use of phosphates alone or mixed with other reagents. Stabilising agents such as sodium laurate may be added to the emulsion. W. L.

41. Improvements In and Relating to Structural Roofing and Like Materials. R. Shaw and Wailes Dove Bitumastic, Ltd. E.P. 300,531. Nov. 12, 1928.

This invention aims at producing a self-supporting roofing material of a bituminous or similar nature. A sheet of structural roofing comprises a metal reinforcement enclosed between two or more sheets of bituminous or resinous fabric, the whole being amalgamated into a strong self-supporting sheet by the application of heat and/or pressure.

The metal reinforcement is composed of rigid interwoven wires, expanded metal, or other perforated sheet metal material, and the waterproofing material of canvas, felt or other suitable fabric coated and impregnated with a bituminous, resinous or similar material. W. L.

42. Improvements in Bituminous Compositions for Use as Supports Exposed to High Pressures. R. Stein. E.P. 299,208. Oct. 25, 1928.

The bituminous composition used for supports consists of epure (natural Trinidad asphalt freed from earthy matter, having a high elasticity, a melting point between 150° C. and 180° C. and 36 to 40 per cent. ash), goudron (a distillation product from Trinidad asphalt). It is of the nature of fatty asphalt, having a melting point between 95° C. and 100° C. and great toughness) and pitch. A preferable combination is one part of epure, two parts of goudron, two parts of coal tar pitch and one part of asbestos fibres. The bituminous materials are brought to boiling point and the asbestos fibres then added. The degree of toughness can be increased by increasing the proportion of asbestos fibres.

This mass is preferably combined with one or more layers of thin wire wound in the form of helices which can be made to mesh if required.

The thickness of the wire may be quite inconsiderable (0·5 mms. to 1·0 mms.), and the diameters of the helices may amount to 3 or 4 mms. The wires are disposed more or less transversely to the direction of motion of loads over the supports. W. L.

See also Abstract No. 16.

Analysis and Testing.

43. Methods to Determine Vapour Pressure. Vapour Pressure Committee, California Natural Gasoline Convention. *Oil and Gas J.*, Vol. 27, No. 32, p. 186.

The object of this paper is to present the results of recent laboratory work on vapour pressures and to show the present trend of that work. The vapour pressure of a liquid mixture is theoretically defined as the pressure in a closed vessel containing the liquid and an infinitesimal amount of its vapour in a state of perfect equilibrium. Two standard methods are described in detail, as references with which to compare practical tests. One, the method of analysing the liquid and calculating the true vapour pressure from the analyses, the other, the use of an extremely large bomb (capacity about 170 litres) with a negligible vapour space which puts the above definition of vapour pressure into physical shape.

Five different methods for the determination of vapour pressures are briefly surveyed and compared with each other and, as far as possible, with the absolute standards described. These methods are (1) Bureau of Explosives, (2) Beistle-Prather, (3) Reid, (4) Gard, (5) Wade Campbell. Of these, the Bureau of Explosives appears the most consistent, but even this method is sensitive to slight variations in procedure. While it is believed that the principle of allowing only negligible evaporation is correct, it is felt that there is insufficient evidence to warrant the recommendation of any one particular method. W. L.

44. Auto-Ignition Temperatures of Liquid Fuels. O. C. Bridgeman and C. F. Marvin, Jr. *Ind. Eng. Chem.*, Vol. 20, No. 11, p. 1219.

Four type methods (the bomb, the crucible, both dynamic and static, the dynamic tube and the method of adiabatic compression) are postulated, and the work of about 30 observers is discussed with respect to certain experimental variables. These include material and volume of the ignition chamber, concentration of oxygen in the inflammable mixture, time lag before ignition, pressure, and the composition of the fuel. Certain trends are noted, but in

general the values reported vary with the specific method and apparatus used. Details are given of a bomb method used by the Bureau of Standards, but the results on gasolines obtained by it are no exception. Analysis of the mechanism of ignition indicates that heats of reaction and heat transfer are of fundamental importance in the interpretation of ignition temperatures.

It is pointed out that the usual methods of measuring ignition temperatures involve considerable time lag, and that any such method, if standardised, would serve to indicate the relative fire hazard from spontaneous combustion. A "true ignition temperature" is defined as the minimum temperature which must be reached by instantaneous heating of an element of volume within a homogenous combustible mixture to initiate inflammation or explosion instantly, and suggestions are made for its experimental determination. When a charge is adiabatically compressed the heating is uniform throughout the charge, and the resulting mean temperature can be computed from the initial conditions and the compression ratio. Ignition in a mixture immediately after adiabatic compression should permit a close approximation to the true ignition temperature as defined. This temperature is believed to be the auto-ignition temperature of most interest in connection with automotive fuels and their relative tendency to pre-ignite and detonate. W. L.

45. Design Visible Distillation Apparatus. R. H. Espach. *Oil and Gas J.*, Vol. 27, No. 24, p. 120.

This paper deals with a pipe-still and bubble tower distillation apparatus for laboratory use.

The pipe-still unit is made of transite board reinforced with small angle iron and is heated by three large gas burners firing directly into the combustion chamber. The pipe coil of ½ inch tube has an internal heating surface of about 1 square foot. The hot gases pass downwards over it and thence to the stack. The inside surface of the furnace is thickly painted with water-glass and covered with asbestos fibre. In the bubble tower the plates, made of stainless steel, are ⅛ inch thick. Each has one bubble cap, the slots in the cap being ¼ inch high. Each plate is drilled to provide an oil draw-off and an oil inlet and is fitted with a down flow tube arranged to keep a ¼ inch layer of condensate on the plate. The plates are separated by glass sections 2 inches outside diameter, and these sections have ground ends which fit into grooves on the plates, vapour-tight joints being assured by the insertion of gaskets between the glass and steel. The whole is bolted together by the rods which pass through holes bored in the plates outside the glass. The tower is insulated by an asbestos lined brass jacket, on opposite sides of which windows are cut and closed with mica to allow of inspection.

The necessary condensers are made of ¼ inch steel tubing bent into a conical helix.

The bubble tower is carried on an evaporator, in which is incorporated a reboiler. The evaporator, 3 inches diameter and 7½ inches high, is divided into two compartments by a horizontal plate. Hot oil from the pipe-still passes through a closed coil in the lower compartment and then discharges into the upper, the liberated vapours passing directly into the bottom of the tower. The residual oil accumulates to a depth of 1½ inches and then overflows into the bottom compartment, where the heated closed coil distils off the more volatile components which are returned to the upper chamber. In the reboiling chamber an oil depth of 1½ inches is maintained by regulating the amount of oil drawn off as bottoms. Small reboilers can be fitted to the tower at any point by inserting a special section. These reboilers are U-shaped steel tubes. The oil from one plate of the column flows through this tube, where it is heated in the up-flow leg by a small electric heating coil, and is

delivered on to the plate immediately below the one it left. The plate on to which this oil is delivered has a ½ inch riser, but no bubble cap.

Quantities between 0·4 and 0·8 gallons per hour can be handled and the temperature differential between the evaporator and the overhead vapour may be varied over a fairly wide range. Tower streams can be taken from the drilled plates and regulated by control cocks.

The writer gives distillation curves obtained on various runs, and it would seem that the reboilers are exceptionally effective, as a tower of seven trays and one reboiler gives practically the same results as a tower of 12 trays.

W. L.

46. Improved Laboratory Rectifying Column. M. J. Marshall. *Ind. Eng. Chem.*, 1928, **20**, 1379.

The column (packed with beads and suitably lagged) is provided with a short side-tube at the level of the thermometer bulb. This side-tube is bent vertically upwards close to the main part of the column and is fitted with a reflux condenser attached preferably by means of a ground-glass joint. On the lower, external curve of the upward bend of the side-tube a slight depression is blown, a run-off tube with stopcock being fitted at this point. This arrangement avoids the hold-up of reflux inherent in the usual apparatus for totally condensing the vapour and returning part as reflux, and gives easier regulation of reflux than the arrangement whereby part only of the vapour is condensed for reflux; moreover, the thermometer can be inserted in the usual manner.

W. S. N.

47. Improvements in Glass Bulbs for Hydrometers and Like Instruments. F. F. W. Flaig. E.P. 302,034. Dec. 13, 1928.

The invention claims to strengthen instruments of the flat or oval type so as to resist expansions and contractions due to change of temperature, etc., by inserting a tube or bridge between the flattened surfaces of the buoyancy chambers.

W. L.

See also Abstracts Nos. 15 and 64.

Refinery Plant.

48. Pipe Stills. D. G. Brandt. *Refiner*, 1928, 82.

Distillation equipment has passed through many stages from the old cheese-box and cylinder stills operating batch with no towers, then connected together for continuous operation, then the addition of towers, and later with pipe-still preheaters. The fractionating towers used to-day are similar to those used in alcohol and benzol fractionation and much of the tower design had already been worked out. This was not true of the pipe-still heater and this part of the unit had to be designed and built from very meagre data. These heaters were originally constructed with ordinary lapweld pipe set in a brick box. The tube bundle was set directly over the combustion chamber. The result was burned out tubes from flame impingement, overheating and oxidation. Dutch ovens were then added, which reduced the tube failures. To overcome flame impingement and small combustion space the tube bundle was placed alongside the fire-box and the flue gases passed downward over them. This also allowed the oil to be passed upward through the tubes countercurrent to the flue gases and increase the efficiency of the heater. This heater worked fairly satisfactorily so long as the fire-box temperature was kept low, but oxidation of the top tubes was rapid and tube failures frequent.

It was very apparent that the arch and back walls reflected considerable heat to the top tubes and in order to reduce this, tubes were installed directly above the tube bundle and up the back wall to absorb this heat. Little or no trouble was experienced if the fire-box temperature was held below 1800°; but the efficiency was still low. Now a complete engineering survey was made checking the B.T.U. transfer for each row of tubes. The transfer rate was found to be very low for the tubes of the bundle and particularly so in the bottom rows, while the tubes in the roof and up the back wall above the bundle were doing more work than the tubes of the bundle, with the exception of the top row. The heaters were then redesigned, the roof tubes being extended over the entire roof and up the back wall above the tube bundles, the air was preheated, and the flue gases recirculated back into the fire-box to reduce the fire-box temperature and increase the gas velocity and temperature difference over the tube bundle. This materially increased the capacity per square foot heating surface over previous designs. These heaters operate with a fire-box temperature of 1400°, stack temperature of 375° and oil inlet temperature of 750°, the thermal efficiency being about 80 per cent. Later developments of this heater have been to again increase the radiant heat absorption surface by using a thin diaphragm to separate the tube bundle from the combustion chamber or installing a baffle behind the first row next to the combustion chamber. Other modifications are to lengthen the flame travel before adding the recirculation gases and rearrangement of ducts and hot fans. Heaters are usually classified as open radiant, shielded radiant and recirculation. Either radiant type is very satisfactory when used where oil inlet temperature is low or where only a moderate air preheat is used. Recirculation heaters are used where high inlet oil temperatures are encountered and usually in conjunction with air preheaters.

Inasmuch as the heater is only a part of the distilling unit and supplies only the heat that is now recovered from the process, it becomes of less importance as the efficiency of the heat recovery increases. The present bubble cap tower is very inefficient from a heat standpoint. Thermally it is a piece of apparatus which takes heat in at a high temperature and delivers it back at a lower temperature, and the longer the range of products produced from a single tower the greater the reduction in temperature between the inlet and outlet of the tower. Take, for example, a single tower pipe-still unit running on crude to a low percentage bottom and taking off gasoline at the top and other products down to wax distillate off the side, through stripping sections. The tower will receive the total crude from the pipe-still at a temperature between 700° and 800° and deliver gasoline vapours at the top to approximately 325°. The side streams will be withdrawn below their boiling points due to steam stripping and it will be necessary to pump back into the tower sufficient gasoline as reflux to absorb all the heat of vapourisation of all products other than the gasoline and all the sensible heat between the inlet temperature and the temperature at which all products leave the tower. Thus we have an enormous quantity of heat at 325° which is far in excess of what the crude can absorb in the vapour exchangers. The interchangers on the side streams and bottoms can recover some heat, but as a whole the crude seldom reaches 400° with very little vaporization due to the high pressure on the interchangers.

To overcome this disadvantage "interchange towers" have been developed. This tower differs from the straight bubble cap tower in that the crude is passed through interchangers between each bubble tray with a fixed temperature differential between the crude and vapours ascending up the tower. Interposed between the vapour interchangers, the crude is passed through the liquid product interchangers which assist in maintaining the proper

thermal gradient. The crude is carried through the interchangers at substantially the same pressure as that in the tower and at a pressure below that of the liquid interchangers. This has a marked advantage over the usual system, as no products are thrown off test by contamination, but any leakage is merely added to the crude and reprocessed. The theory on which the tower operates is entirely different from the straight bubble cap towers and is that of fixed temperature gradients between the trays. Although the reflux flows downward from tray to tray it increases in quantity as it proceeds downwards unless taken out by the proportional reflux boxes. The side streams are taken out and handled similar to other towers except that the kerosine is made as an overhead redistillated product. The crude leaving the last interchanger has been heated to 500–550°, and approximately 45 per cent. of the crude has been vaporised. At this point the vapour is separated from the liquid and the liquid is pumped through the heater and into the separator where the vapours and liquid are separated, and the vapours passed to the base of the tower along with those previously removed from the crude. The only water coolers used are those on the kerosine and final gasoline coolers. Both the thermal and fractionating efficiencies are extremely high. The control of the unit is simple and the only automatic equipment necessary is the flow controller on the crude supply. S. W. C.

49. Process and Apparatus for Fractionating Hydrocarbon Oils. J. C. Black. U.S.P. 1,692,072. Nov. 20, 1928.

This invention is a continuation of a previous application, Serial No. 630,147, and describes a process for separating fractions of any desired boiling range from mixtures of hydrocarbons, and embodies improvements which simplify the apparatus, reduce labour and economise heat for evaporation. Two fractionating columns used in series are employed, the operation being conducted in two stages; if, however, closed fractionation is desired more columns may be employed. The apparatus is well adapted for the production of gasoline from crude oil or naphtha, and when operating on crude oil it will produce a finished test gasoline, which does not require subsequent steam distillation. Hydrocarbons of specific boiling point may be introduced into the apparatus, where they are blended in the vapour state with the existing vapour from the crude, in order to produce a homogenous mixture which will yield a final condensation product of any desired boiling range. Intermediate fractions of any desired boiling range between gasoline and heavy steam still residue may also be obtained.

Diagrams of the apparatus, together with a detailed account of the method of operating the series of fractionating towers, are given. H. E. T.

50. Improvements Relating to Heat Exchangers. Compagnie des Surchauffers. E.P. 292,070. Oct. 4, 1928.

This invention relates to heat exchangers of the type in which one element through which one fluid flows, is arranged within a casing through which the other fluid circulates. The delivery pipe from the casing space is a syphon tube, the legs of which are of equal length. At the lower ends of both legs are cups which always contain liquid into which the pipes extend. By raising or lowering the syphon pipe the level of the liquid in the casing can be altered at will, thus allowing a large variation in the heat transference.

The syphon pipe is of a cross-section sufficiently greater than that of the inlet pipe to the casing as to ensure a sufficient out-flow speed at all times relative to the speed of inflow to prevent a rise in liquid level in the casing.

The syphon pipe may be counter-balanced and its movements controlled by a thermostat to give constant temperature delivery of either of the fluids passing through the exchanger. W. L.

51. PRODUCT OF FRACTIONATING. Ira B. Funk. U.S.P. 1,691,238. Nov. 13, 1928.

The main object of this invention is to provide a fractionating process and apparatus, for separating desired fractions from hydrocarbon vapours. The apparatus yields close cuts as regards boiling range from a mixture of hydrocarbons obtained by steam distillation; the fractionating tower being provided with a series of water-traps for separating out water from the vapours. The process is applicable to all classes of treatments for hydrocarbon products where steam is used. The main improvement consists in the form and method of operation of the fractionating column which is constructed as follows:—

The column is provided with a series of perforated plates through which pass a series of funnels for passing reflux condensate back to lower plates in order to dephlegmate rising vapours. In the upper part of the tower are placed "water plates" having passages through them covered by bubble caps. Leading from each water plate is a line passing to a water-trap, where both water and condensed liquid are drawn off. This condensed liquid is returned to the column at a point below the water plate, from which it was drawn off, while any uncondensed vapour is returned to the column at a point above the plate. Close cuts off desired boiling point are obtained, when steam is used during the distillation, by eliminating the partial pressure effect due to reboiling water in the column. This is accomplished by drawing off the condensed steam at the proper level in the column. The capacity of the column is increased by progressively decreasing the areas of the orifices in the plates. The average pressure under which the column is worked is 40 lbs. per square inch. H. E. T.

52. IMPROVEMENTS IN DEVICES FOR AUTOMATICALLY CLOSING THE GAS SUPPLY ON EXTINGUISHING OF THE BURNER. T. Herszlik. E.P. 291,309. May 31, 1928.

The invention provides for a device of the type in which a pawl or brake actuated by a heat-responsive expansible member is arranged in proximity to a locking wheel connected with the gas valve. The expansible member consists of a one-part thin member, such as a wire or strip, capable of resisting very high temperatures, and is subjected at its free end to tensile stress by means of a spring for the purpose of preventing the possibility of its bending or sagging. All parts serving for the locking or releasing of the gas valve, with the exception of the expansible member itself, are arranged inside the valve casing for the purpose of ensuring correct operation of the device, its inaccessibility from outside and its remaining clean. The wire or strip subjected to the heat is fixed at its upper end to a sheet metal shield which is arranged parallel to the burner tube, so that the upper part only of the wire or strip projecting from the sheet metal shield is heated by the flame of the burner, until on the burner becoming extinguished the heating operation ceases. S. W. C.

53. IMPROVEMENTS IN AND RELATING TO FRACTIONATING TOWER. Power Speciality Company. E.P. 270,720. Nov. 5, 1928.

An improved fractionating tower is described in which bubble trays are employed for separating liquid and vapour, with the separate recovery of fractional condensates of any desired boiling range.

The fractionating tower consists of a vertical cylindrical casing, fitted with a series of bubble trays arranged in groups and supported by the casing in a liquid-tight manner. At the bottom of the tower is a residuum outlet, and somewhat above this is an oil and vapour inlet, while at the top of the tower is a vapour outlet. Means are provided for uniformly distributing the

liquid over the bubble trays, and an arrangement is located beneath each tray, for uniformly distributing the rising vapour and separating entrained liquid from it. Each bubble tray is fitted with a drain and a fractional condensate outlet in the casing, the latter being provided with an adjustable trough, by means of which the whole or a variable fraction of the liquid leaving one tray or group of trays may be withdrawn. The oil and vapour inlet at the bottom of the tower is arranged tangentially, causing incoming oil and vapour to impinge against a perforated baffle spaced apart from the casing which produces an initial separation of liquid from vapour. Between the oil and vapour inlet and residuum outlet at the bottom of the tower is a series of bubble trays forming a liquid seal, and preventing vapour coming in contact with the heavy liquid which collects in the bottom part of the tower. The tower is provided with a series of fractional condensate outlets located beneath alternate trays, these outlets being arranged in groups at different levels, each group having a common delivery duct, and each outlet a valve controlled pipe connecting it to its corresponding duct. Beneath each bubble tray is a vapour-distributing and liquid-separating screen provided with a series of inclined parallel blades arranged at right angles to the elongated vapour ports in the tray. At the lower edge of each blade are inclined gutters and troughs for collecting liquid and conveying it to the supply trough of a subjacent bubble tray.

The construction of the tower possesses important practical advantages, especially in its flexibility to yield fractional condensates of widely different quantity and character. The tower is well-adapted for use under widely varying conditions, and moreover has a relatively large capacity. H. E. T.

54. Improvements in and Relating to Bubble Still Trays for Fractionating Towers. Power Speciality Company. E.P. 299,479. Nov. 5, 1928.

This invention describes improvements in bubble-still trays for use in fractionating columns. The bubble-still tray consists of a series of sections, with fluid tight joints between each section, and a series of vapour ports in each section, with superposed bells co-acting with each upright port, to form a liquid seal. Flanged troughs are placed at the end of each section and port, with liquid seals between the troughs and sections. The liquid is uniformly distributed over the sections by means of a supply weir, a discharge weir being used for controlling the depth of liquid in the sections, and delivering it into the second trough. A drain is placed in the second trough for conveying liquid to a subjacent tray or fractional condensate outlet in the fractionating column. H. E. T.

55. Oil Heater. John Primrose. U.S.P. 1,693,155. Nov. 27, 1928.

The apparatus comprises a number of tubular elements and end connections which provide a plurality of similar flow paths for the oil to be heated. Each path consists of a number of the elements in series, a supply member into which the inlet end of the initial element of each series is connected, and controlling means for each flow path. G. S.

56. Refining Oils, Apparatus for. John Primrose. U.S.P. 1,693,156. Nov. 27, 1928.

The heating furnace consists of a fire chamber and a tube chamber, the latter being separated from the fire chamber by a bridge wall over which the heating gases pass and leave by an outlet at the bottom of the tube chamber. By means of a circulating pump, the oil is circulated from an external tank through the tubes at successively higher levels and back to the

tank. The heating gases from the base of the tube chamber may be passed under the tank and means may be embodied for recovering heat from the evolved vapours. Means are provided for supplying additional untreated oil to the tank. G. S.

57. An Improved Method of and Means for Sealing Floating Roofs of Liquid Storage Tanks. Standard Oil Development Co. and Conrad Arnold. E.P. 298,010. Oct. 4, 1928.

According to this invention a floating roof for a tank comprises a roof plate having an upper rim fastened to it, a flexible inflated member below this rim and a floating member below the inflated member for distorting the latter and so producing a sealing effect between the floating roof and the side walls of the tank.

The roof plate is a flat circular plate adequately strengthened and of smaller diameter than the shell of the tank. Rigidly fastened round the perimeter of the roof plate is a steel ramp extending upwards and curving outward at the top to form an upper rim. This rim is annular in shape, extending round the roof and to within a few inches of the side walls of the tank. Near the tank wall floats are regularly spaced round the perimeter of the roof plate and float in the liquid contained in the tank. Supporting arms are fastened to the upper surfaces of the floats and support a lower curved plate. The upper curved rim and the lower curved plate are respectively concaved on the lower and upper sides and so adapted to hold a permanently inflated annular member between them. This inflated member is made of canvas impregnated or coated with rubber or similar impervious material, and when in position is under sufficient pressure to cause it to form a seal between the roof and the wall of the tank. This member may be in one piece or, preferably, of several non-communicating fluid chambers. The lower curved plate is pressed upwards by the buoyant force of the floats and thus the flexible annulus is distorted from its normal circular cross-section and is pressed tightly against the side walls of the tank and the concave under side of the upper rim, thus forming a tight sliding seal. W. L.

58. Distillation. George W. Watts. U.S.P. 1,689,606. Oct. 30, 1928.

The oil under treatment is introduced to a mixing device, together with superheated steam under reduced pressure. The steam and oil mixture passes through an unheated passage until equilibrium is attained at a predetermined temperature and pressure without separation. The unseparated liquids and vapours are separately withdrawn from a chamber. G. S.

See also Abstracts Nos. 13 and 19.

Engineering.

59. The Effect of Hydrocarbon Vapour on the Contact Points of Ignition Apparatus. E. A. Watson. *Auto. Engineer*, Vol. 18, No. 245, p. 347.

Manufacturers of ignition apparatus are familiar with occasional cases where the contact points fail after very short periods of use. The normal life of such points is at least 20,000 miles, but the occasional failures may be repeated on replace points after an abnormally short life, especially with platinum contacts, and even replacement of the whole ignition unit does not remove the trouble.

It is found that the difficulty is prevalent when the magneto is exposed to hydrocarbon vapours, whether from petrol drip or from the crank-case vent valve.

The effect of a condenser in suppressing a spark at the contact breaker points is not due to the relation between the rate of voltage rise and the rate of separation of the points. The criteria of arcless break are the current flowing through the contacts and the surface conditions existing at the time of separation.

The minimal arc characteristics when a condenser is shunted across a gap are dealt with mathematically and it is shown that the arc current may either cease abruptly in less than a micro-second, or be prolonged in wave form for an indefinite time, according to the inductance of the local circuit.

To investigate the question experimentally a standard contact breaker was mounted in an air-tight chamber so that behaviour could be observed in various vapours. Petrol, benzol and ethyl alcohol were used and in all cases the contact points were attacked much more rapidly than in air. Alcohol gave the least wear. Carbon deposit surrounded the points after each test in vapour. Platinum points failed rapidly, whereas tungsten points were comparatively lightly attacked.

It is concluded that some arcing takes place under working conditions with currents as low as 0·5 amperes, and that the deposition of carbon alters the condition of the break, permitting sustained instead of quenched arcing. In the case of platinum points the effect is more destructive on account of reaction between the film of carbon and the metal. The trouble may occur in any engine, irrespective of fuel, if the contact breaker is exposed to vapour from the carburettor system or from the crank-case breather. R. S.

60. Report on Heavy Oil Engine Working Costs. Diesel Engine Users' Association. Dec. 7, 1927–28.

Details of conditions and working costs are tabulated and annotated.

Table 1 gives a list of engines dealt with in order of type and power, and includes average age, hours run, and average running plant load factor.

Table 2 gives engine costs per unit generated at each of 51 stations for the year 1927-28. The total costs are analysed and include prices paid for and the consumption of both fuel and lubricating oil.

Table 3 gives average costs per unit generated at each of 23 stations over a period of six years.

The 1927–28 returns show a slight addition to the number of mechanical injection engines. The overall results for home stations are about 3 per cent. better than for the previous year for approximately the same average fuel and lubricating oil prices.

The improvement is attributed to better running conditions, the annual plant load factor and running plant load factor being higher.

The overall cost for overseas stations is unchanged, the decrease in running costs being offset by an increase in the average price of fuel oil.

The average engine cost per unit generated, including fuel, lubricating oil, stores and water, wages, and repairs and maintenance, varies from 0·741 pence for very old engines to 0·641 pence for mechanical injection engines.

There is little to choose between normal types of air injection engines and high compression airless injection engines.

The total output reported upon has increased by 11·8 per cent. on the previous year's output and the new plant installed is 3,510 kw.

The overall thermal efficiency determined from the gross calorific value of the fuel used and the units generated is 24·66 per cent. The average fuel

consumption per unit generated is 0·71 lb., and the average lubricating oil consumption is 0·99163 gallons per unit generated.

Cleaning of used lubricating oil is receiving special attention, the centrifuge, the stream line filter and settling methods being in use.

Repairs and maintenance average 0·108 pence per unit for home stations.

Information is particularly required regarding operation of peak load installations and stand-by service plants in connection with bulk supplies, and also regarding the working conditions of cylinder liners which have had an unusually long life. It is suggested that long life of cylinders and cylinder-heads is dependent on avoidance of scale deposits in the water spaces.

R. S.

See also Abstracts Nos. 42, 57, and 97.

Anti-Detonation.

61. Explosions in Closed Cylinders. Part I. Methane-Air Explosions in a Long Cylinder. Part II. The Effect of the Length of the Cylinder. W. A. Kirby and R. V. Wheeler. *J. C. S.*, December, 1928, p. 3,203.

The object of this research is the correlation of the movement of flame, with development of pressure in a cylinder whose length is great compared with its diameter.

Part I.—The cylinder employed was 200 cms. long, 10 cms. internal diameter, having a capacity of approximately 16 litres. For ignition at one end of the cylinder, the time taken by the flame to travel throughout its length corresponded with the time of attainment of maximum pressure for all the mixtures of methane and air studied. The mean speed of the flame is considerably faster than that produced by central ignition in a sphere of 16 litres capacity. At the moment of ignition at one end of the cylinder the flame darts forward with rapidly increasing velocity; a retardation period then ensues until the flame is about 20 cms. from the other end of the cylinder, when it moves forward with increasing velocity. From the photographic records obtained, it is seen that the flame assumes a vibratory character at the end of its travel, although vibrations sometimes occur after the flame has travelled half-way along the tube. In a long cylinder with ignition at one end, the flame touches the walls long before the propagation is complete and hence cooling of the products of combustion begins at an early stage. As a result of this cooling the rate of development of pressure and maximum pressure attained are lower than those for central ignition in a sphere of equal capacity. The time-pressure records were of two types, depending on the composition of the methane-air mixture employed. For mixtures containing less than 8 per cent. or more than 11 per cent. of methane, the curves were smooth, but for mixtures containing 8–11 per cent. of methane the curves showed marked vibrations at their crests. The time-pressure and time-distance curves show that the development of pressure corresponds closely with the movement of the flame. When the point of ignition was varied, the maximum pressure recorded occurred when the ignition was at the centre of the cylinder and this pressure was higher than that for ignition at one end. A series of experiments were also made in which methane air mixtures containing 9–10 per cent. of methane were ignited at some point along the axis of the cylinder and at one end simultaneously. From the results obtained it was found that the rate of rise of pressure became more rapid as the second ignition point was moved towards the centre of the cylinder. The initial rate of development of pressure was

then more rapid than for ignition in any single position. The maximum pressure with a double ignition occurred when ignition was effected at one end and at the middle of the tube simultaneously, but this pressure was lower than that for a single ignition at the centre of the tube.

Part II.—The critical length of cylinder for the production of a vibratory explosion with a 9·5 per cent. mixture of methane and air was found to be 140 cms. With tubes of shorter length, the movement of flame and the time of attainment of maximum pressure corresponded closely, the rate of rise of pressure being regular. When the tube was 140 cms. or more long, rapid vibrations were usually developed by the flame towards the end of its travel, and synchronising with these vibrations the time-pressure curves showed rapid fluctuations at their crests. These rapid vibrations were always accompanied by a shrill screech. Similar results have been obtained by Maxwell and Wheeler (*J. Inst. Pet. Tech.*, 1928, **14**, 175) for pentane-air mixtures.

For a given explosion mixture of methane and air it appears reasonable to suppose that the establishment of resonance in the column of gas is due to the rapidity with which the column is compressed. This explanation is, however, shown to be insufficient, and the authors believe that the character of the reactions occurring behind the flame front have an important bearing on the production of resonance. H. E. T.

62. Explosions in Closed Cylinders. Part III. The Manner of Movement of Flame. D. C. de C. Ellis and R. V. Wheeler. *J. C. S.*, December, 1928, p. 3,215.

Various interesting photographs are published showing successive stages of the propagation of flame, produced by ignition in a mixture of 10 CO + O_2 saturated with water vapour. A comparison is drawn between the manner of the movement of the flame in carbon monoxide-oxygen mixtures and in methane-air mixtures. (Parts I. and II., *J. Chem. Soc.*, December, 1928, p. 3,203.) H. E. T.

63. Flame Movement in Gaseous Explosion Mixtures. O. C. de C. Ellis. *Fuel*, 1928, Vol. 7, pp. 502–508, Part 8.

An account is given of the propagation of flames in cylindrical tubes from ignition effected at one end. The flame thus formed resembles a half of that produced by central ignition. In this type of ignition cooling produced by conduction to the walls of the tube commences at the moment of ignition, the loss of pressure at any moment due to this cause being proportional to the area of the cooling surface and the pressure in the tube. Interesting photographs are published showing the progress of the flame after such an ignition, together with those produced by ignition in a series of tubes of varying lengths but accurately equal diameters.

An account is given of the movement of flame produced (1) by ignition in tubes closed at firing end only and (2) by ignition in tubes open at firing end only. H. E. T.

See also Abstract No. 44.

Cracking.

64. Critical Temperatures and Oil Cracking. R. H. McKee and H. H. Parker. *Ind. Eng. Chem.*, 20, 1169.

This investigation was carried out to determine the critical temperatures of complex mixtures such as gasoline or gas oil with a view to using them as

a basis for differentiation between liquid and vapour phase cracking. It is shown that these complex mixtures have definite critical temperatures, and that the critical temperatures can be calculated from the average boiling points obtained from the ordinary standard analysis (A.S.T.M. method) by the following equation :—

$$t_c = 1{\cdot}05t_b + 160$$

where t_c = critical temperature in degrees Centigrade,
t_b = the average boiling point.

This represents the relationship between the boiling points and critical temperatures of normal straight chain aliphatic hydrocarbons, also this equation holds for mixtures of straight or branched chain saturated and unsaturated aliphatic hydrocarbons and for naphthenes, but not for aromatic hydrocarbons.

The work also revealed the fact that the critical temperatures of oils used as cracking stock were lower than the temperatures used in some of the more important liquid phase processes, such as the Cross and the "Tube and Tank." In the operation of these, the temperature of the oil leaving the cracking tube is about 454° C. The critical temperature of the charging stock is generally less than this, and is lowered as cracking proceeds, owing to the formation of lighter products.

Experimental work with mixtures of gas oil and gasoline confirm the lowering of the critical temperature, and such mixtures appear to obey the rule of Straus and Pawlewski, which states that the critical temperature of the mixture is proportional to the volume per cent. of the components.

With regard to aromatic compounds the relationship may be expressed thus :—

$$t_c = t_b + 208$$

This equation should give the true critical temperature of a mixed distillate only if the oil contained 100 per cent. aromatic compounds. The first equation should hold if no aromatics are present. In a case where both types of hydrocarbons are present, the critical temperature will be intermediate between calculated values by the above equations. This critical temperature work may thus be used to indicate the chemical nature of the oil.

Finally, work with solutions of asphaltic material in oil showed that no precipitation took place even when the liquid was kept for a long time above the critical temperature, thus proving that the asphalt was soluble in the highly-compressed vapour.

The apparatus in which the work was carried out consisted of Pyrex glass tubes 10 to 12 cm. long, which were sealed by using an oxy-gas flame whilst the filled end of the tube was immersed in a freezing mixture. The tubes were heated in an electric furnace and the critical temperature, the point at which the meniscus disappeared on heating and appeared on cooling was measured by a platinum, platinum-rhodium couple in conjunction with a portable potentiometer. R. P.

65. Liquid-Phase Cracking Processes Show Modern Developments E. Owen. *Chem. and Met. Eng.*, 1928, **11**, 677.

The paper is the first part of an article wherein the author discusses the basic principles of liquid-phase cracking, and describes shortly the older processes, giving various facts about the history of liquid-phase cracking and the processes connected with the development of the modern cracking plants which are replacing the older units, on account of their greater operating efficiency, larger yield of gasoline and larger daily capacity.

The Burton, Coast, Fleming, Jenkins, Isom and Dubbs processes are compared in this part of the article, and the Cross, Holmes-Manly and Tube and Tank processes will be discussed in a subsequent paper. R. P.

66. Process for Cracking Oils. J. G. Alther. U.S.P. 1,685,476, 1928. June 9, 1926.

The oil to be cracked is pumped through heating coils of progressively decreasing diameter, the widest coils being placed in the hottest part of the furnace, so that the oil attains its highest velocity in the coolest furnace zone. The product then enters a vapour chamber from which gasoline and recycle oil are separately withdrawn. H. G. S.

67. Conversion of Hydrocarbon Oils into Lighter Oils. C. Arnold. E.P. 299,683. Sept. 22, 1927.

A cracking plant consisting of a heated coil and digestion drums, connected so that one may be used while another is being cleaned, is improved by filling the drum or drums to be brought into use with heated oil from the coil. Oil from the drum or drums to be taken out of use may be used with the heated oil from the coil. G. S.

68. Art of Cracking Hydrocarbons. J. E. Bell. U.S.P. 1,688,325. Oct. 23, 1928.

A battery of vertical cracking tubes in parallel are heated by hot gases from a furnace so that the gases travel downward counter-current to the oil flow. The oil is pumped by means of a circulating pump from the bottom of a still into the bottom header of the cracking tubes; the oil flows upwards through these tubes into another header connected to a tube which discharges into the still at the end furthest from the pump suction, and some distance above the bottom of the still.

The oil coming from the bulk supply is thus heated to cracking temperature as it flows upward through the cracking tubes, being progressively heated to maximum temperature in the direction of its flow.

The process may be carried out in a still in which the circulation is maintained by thermo-siphonic action, but by means of the pump an increased rapidity of circulation can be maintained, with improved heat transfer and greater protection of the tubes. R. P.

69. Operation of Pressure Stills. J. E. Bell. U.S.P. 1,693,946. Dec. 4, 1928.

In cracking oils for the production of gasoline by distillation in pressure stills provided with reflux towers, it has been customary to condense the vapours to give a pressure distillate, then to re-distil this distillate to separate the gasoline.

This method involves a loss of heat and, to dispense with the redistillation, the vapours from the still are caused to pass counter-current to a stream of raw oil in a zone separate from the vaporizing zone, thus preheating the raw oil and separating the distillate into a series of fractions.

The reflux and charging stock return to the still and the vapours are subjected to a fractional condensation in a series of condensing chambers in indirect heat exchange with the incoming raw oil. A part of the raw oil employed in the fractional condensation is introduced into the refluxing operation and another part into the still.

A pressure still, a reflux tower and a fractional condenser of special design, consisting of a series of condensing chambers maintained at progressively lower temperatures, are utilized to obtain the results specified. R. P.

70. Art of Converting Hydrocarbon Oils. O. C. Brewster. U.S.P. 1,694,092. Dec. 4, 1928.

Air or other oxidising gas is supplied to a still at its lowest part and the heat required for the conversion of the oil is supplied by internal combustion. Suitable means are provided exterior to the still for bringing the contents to a temperature at which oxidation will take place. A heating coil connected to the still is suitable for this purpose and a pump produces positive circulation through the coil.

Means are also provided for supplying oil to the still, after the operation has begun, in the form of a feed preheater which consists of a bubble tower. Fresh oil is pumped into the upper part of this tower after it has been brought to a temperature at which oxidation will take place spontaneously when air is supplied near the base of the tower. The supply of air is controlled to give the required temperature, 350° C. to 385° C.

The period during which the oil remains in the preheater is so short that practically no conversion takes place and the oil passes into a stripping column counter-current to the vapours rising from the still. The vapours are stripped of heavy ends and the fresh oil, together with the reflux, flows into the still, whilst the gas and vapours go to a condenser and cooler. The condensate is sent back to the preheating column with the feed stock, and the spirit from the cooler is collected. R. P.

71. Continuous Transformation into Light Hydrocarbons of the Heavy Hydrocarbons from Petroleum. Henri Carroll. E.P. 284,345. Jan. 28, 1928. Conv. (France) Jan. 28, 1927.

The heavier portions of an oil are cracked by causing them to flow while hot and in thin layers over a series of steps of increasing surface area and temperature. Crude oil may be subjected to a preliminary distillation in a tube, or other still, from which the liquid products pass to the cracking apparatus. G. S.

72. Apparatus for the Manufacture of Gas from Oils. C. Chilowsky. E.P. 293,008. Oct. 11, 1928.

In prior specifications, Nos. 255,423, 271,899 and 271,907, processes and apparatus have been described which consist essentially of atomising heavy oil, then effecting partial combustion with air or oxygen in such a manner as to obtain a temperature of between 700° and 1000° C. The products pass into a container maintained in a state of incandescence and containing a catalyst by means of which fixed gas is produced.

The present invention comprises two units which can function alternately. Whilst one unit is in use the other may be cleaned of the deposits by the passage of the air employed to ensure partial combustion in the other unit.

The plant consists of a single atomiser to which the oil is led, the oil being atomised first on the right-hand part and then on the left-hand part. At any convenient point steam may be injected into the atomised oil.

There are two outlets and two inlets to the plant and a simple piston valve reverses the current of air as required. R. P.

73. Improvements in the Conversion of Heavier Hydrocarbons into Lighter Hydrocarbons. R. Cross. E.P. 273,256. Aug. 13, 1928.

The plant consists essentially of a heating coil, a reaction vessel, polymer separator and a bubble cap dephlegmator, condenser and gas separator.

The oil is preheated by passing through a coil in the upper part of the dephlegmator. Into the stream of hot oil a pump delivers the reflux from

the dephlegmator and the mixture passes into the heating coil, at a pressure of from 700 to 1500 lbs. Sufficient heat is applied to the coil to complete the process, but not enough to cause excessive coke deposition in the tubes. The oil is then transferred to the reaction vessel, which is formed of a single ingot of forged steel. In commercial plants these chambers may be of 30 to 72 inches in diameter and 30 to 60 feet long. The reaction vessel is not heated, but is heavily insulated to prevent loss of heat. The pressure within this vessel may be from 700 to 1000 lbs. per square inch.

The time occupied in heating the oil is 2 minutes or less and the time of reaction is 10 to 60 minutes.

The pressure is released as the oil leaves the reactor and it expands into the polymer tower, whence heavy residue is drawn off at the bottom and the vapours pass through an overhead line to the bottom of the bubble tower. End-point spirit is obtained and the reflux, as before mentioned, is pumped into the raw oil stream to the heating coils.

By the use of this plant it is claimed that oils having a critical temperature above the working temperature, but a critical pressure below the working pressure, such as gasoline and kerosine, can be cracked to yield lighter hydrocarbons of the paraffin naphthene and aromatic series. Under the conditions the olefins or unsaturated hydrocarbons are converted into cyclic or naphthene compounds, or heavy polymers. These heavy polymers form a pool in the reaction chamber, which serves the purpose of a scrubbing material which accumulates and separates carbon and yields a fuel oil from the polymer separator containing less than 0·5 per cent. of suspended carbon.

Four hundred barrels of anti-detonating fuel motor fuel may be produced per day under the conditions specified. The product is characterised by high ratio between specific gravity and the distilling temperature and at the same time low percentage of unsaturated compounds, these having been largely converted into naphthenic compounds. R. P.

74. Process for Converting Petroleum Oils. A. D. David. U.S.P. 1,688,855. Oct. 23, 1928.

The process consists of cracking gas oil at 375° to 475° C. at pressure of from 50 to 300 lbs. The oil is raised to cracking temperature in a heating coil and transferred to a large reaction chamber. The vapours from the chamber are condensed and the residual oil continuously withdrawn by a pump and discharged back into the chamber in such a manner as to produce a swirling action to the body of oil in the chamber, thus maintaining the oil in a turbulent condition in order to maintain the carbon in suspension in the liquid residue which is drawn off into settling tanks. These tanks may be cut out or in as desired and the residue permitted to collect and separate its solid content. Any number of settling tanks may be used in order to keep the plant running continuously. They are provided with manhole doors to facilitate cleaning. R. P.

75. Method for Treating Hydrocarbon Oils. C. P. Dubbs. U.S.P. 1,686,654. October, 1928.

In this process oil is pumped through a heating coil which is maintained at a suitable temperature. It then passes into a vertical tank into which a regulated amount of raw oil is pumped and the mixture overflows into a comparatively shallow conical bottomed tank wherein carbonaceous matter settles out. Vapours from both tanks pass upward through air-cooled reflux tubes, are condensed in a water condenser, and thence pass into a gas separator.

The settling tank is in duplicate in order to make the process continuous, means are provided for drawing off residuum and free carbon either intermittently or continuously. One tank may be cleaned whilst the other is in use.

A pressure within the cracking coil is maintained sufficient to prevent vaporization, but is reduced in the tanks to permit the separation of the lighter constituents. R. P.

76. Process of Cracking Hydrocarbons. G. Egloff and H. P. Bonner. U.S.P. 1,688,859. Oct. 23, 1928.

In this process coal, shale, or other bituminous substances are dispersed in coal tar in the colloidal condition and the mixture cracked to produce light hydrocarbons.

Fuel oil or heavy Mexican or Californian crude oils may be mechanically mixed with at least 10 per cent. solid bitumen in colloidal form, as, for example, finely powdered bituminous coal, can be injected into the oil in such a way as to form a colloidal dispersion of the bitumen in the oil.

The mixture is distilled by means of heat and pressure in any suitable cracking apparatus.

The pressure distillate produced may amount to from 20 to 60 per cent. of the original charge. R. P.

77. Process for Cracking Oil. G. Egloff. U.S.P. 1,688,860. Oct. 23, 1928.

In this process cracking takes place in two separate zones and the vapours produced in each zone are separately condensed. Different conditions of temperature and pressure may be maintained in each zone whereby additional cracking is effected after passing the uncracked product of the first zone into the second zone. The apparatus consists of a heating coil, a vaporizing chamber, dephlegmator and condenser of the usual type. The unvaporized portion of the oil remaining in the vaporizing chamber is drawn off by a pump and discharged into a secondary chamber, which is subjected to an increased temperature caused by a top-fired furnace.

In this secondary zone of increased temperature and pressure additional cracking is effected and the vapours pass through another dephelgmator into a condenser and collecting vessel. The reflux condensate from the dephlegmator returns by a common pipe to the charging pump.

The temperature of the first zone ranges from 400° to 430° C. with a pressure of from 50 to 200 lbs., whilst the temperature of the second zone may be above 460° C. at a correspondingly increased pressure.

By treating a mid-Continent gas oil in a process of this kind 40 to 50 per cent. of distillate ranging from 48 to 52° Bé. may be produced. R. P.

78. Process of Cracking Petroleum Oil. G. Egloff. U.S.P. 1,688,861 Oct. 23, 1928.

The invention refers particularly to the method of treating residuum produced by cracking.

The plant consists of a heating coil situated in a furnace, a large expansion chamber with overhead outlet to the dephlegmator and condensers. The reflux oil from the dephlegmator returns to the raw oil feed, whilst the vapours pass to the condensers which separate the spirit and gas.

From the expansion chamber the bottom outlet discharges heavy residue through a cooling coil into a treating tank. The residue is introduced into this tank through a perforated pipe which extends along the bottom. The

tank is half full of liquid sulphur dioxide and an agitating device mixes the contents of the tank. After settling, the upper layer is drawn off and neutralized with caustic soda solution, and the bottom layer is distilled to recover the sulphur dioxide.

The upper layer may be suitable for the production of lubricating stock by treatment with sulphuric acid and distilled into suitable fractions. The lower layer on similar treatment may yield good cylinder stock. R. P.

79. Improvements in the Conversion of High Boiling Point Int. Compounds of Lower Boiling Point. I. G. Farbenindustrie A.-Go E.P. 270,313. Sept. 28, 1928.

The invention is an improvement or modification of the process described in E.P. 268,796.

As suitable mixtures for the process, cobalt, nickel, molybdenum, chromium, tungsten, manganese and other alloys are mentioned. It has now been found that titanium, zirconium, niobium, tantalum, thorium, uranium, copper, thallium, boron and silicon, and their alloys may be suitably employed in the said conversion. As an example 5 parts of titanium are added to 100 parts brown coal producer tar, 20 per cent. of which distils up to 300° C. The mixture is heated to 300° C. in a still whereby 50 per cent. or more distils over up to 300° C. R. P.

80. Improvements in the Destructive Hydrogenation of Coal Tars Mineral Oils and the Like. I. G. Farbenindustrie A.-G. E.P. 279,072. Dec. 20, 1928.

In a previous patent specification, No. 254,713, relating to a process for the destructive hydrogenation of coal, etc., use is made of gases prepared from gases containing hydrogen by partial decomposition with oxygen, in the presence or absence of catalysts, with conversion of carbon monoxide into carbon dioxide and hydrogen.

It has been found that oxygen can be replaced by carbon dioxide either alone or in association with steam for decomposing the hydrocarbon gases.

The process may be carried out with the catalysts mentioned in the above specification or with other suitable catalysts. Valuable constituents may be separated from the initial gases prior to the treatment with carbon dioxide and employed for other purposes.

The process may be conducted in a shaft furnace filled with refractory masses which may be catalytically active.

For example, a gas containing 30 to 40 per cent. of methane obtained in a destructive distillation of coal or the like is mixed with an amount of carbon dioxide corresponding to the methane content and passed through the furnace at a temperature of 1100° C. The resulting product is a gas mixture containing 66 per cent. H. 31·4 per cent. CO, 1·1 per cent. CO_2, and 1·3 H. without any appreciable deposition of soot.

By treatment with steam at 500° C. the carbon monoxide may be converted into CO_2, which can be washed out under pressure. R. P.

81. Improvements in the Manufacture and Production of Valuable Hydrocarbons. I. G. Farbenindustrie A.-G. E.P. 302,253. Dec. 10, 1928.

It is known that carbonaceous materials may be converted into saturated products of the character of benzine, which when used in engines with high compression give rise to knocking, by treatment with hydrogen at elevated temperatures under pressure and if desired in presence of a catalyst.

Valuable liquid products may now be obtained from the same carbonaceous materials by carrying out the operation at temperatures above 550° C. and at a pressure of at least 20 atmospheres, with hydrogen in excess. The employment of hydrogen in excess is very important for avoiding undesirable decomposition, particularly the formation of carbon and pitchy substances.

Depending on the nature of the raw material and the temperature of working, hydrocarbons of low boiling point, part of which are unsaturated, are obtained. As a rule the higher the temperature, the lower will be the boiling point and the greater the unsaturation of the products. With initial materials containing hydroaromatic hydrocarbons, there will be a greater tendency towards the production of aromatic hydrocarbons. It is essential that the residues, from which low boiling products have been split off, should be slightly hydrogenated to prevent the formation of coke or pitch.

The conditions of working having a particular influence on the reaction are: the temperature, which must be above 550° C., the partial pressure of the hydrogen, the absence of materials which give rise to deposition of carbon and the formation of methane, and the nature of the raw material. If one of these conditions varies, the others must be adjusted. By the addition of catalysts, either the cracking or the hydrogenation may be accelerated. Elements of the third and fourth group of the periodic system are suitable for the former reaction. Catalysts which possess good efficiency both for cracking and hydrogenation are molybdenum and its related elements.

As an example, the vapours of a heavy middle oil obtained from brown coal tar oil with the aid of cracking catalysts are passed with about twenty times an excess of hydrogen over a chromium oxide catalyst heated to 575°/600° C. at a pressure of 200 atmospheres. The walls of the cracking apparatus are constructed of Krupp's W. T. 2 steel, which does not give rise to the separation of carbon or the formation of methane. Free steel or nickel should be avoided. The gases produced are cooled under pressure and yield a mixture containing 85 per cent. of benzene, 5 per cent. of toluene and the like. The residue consists of naphthalene and unchanged raw material. R. P.

82. Processes for Cracking Hydrocarbons. Gasoline Products Co. E.P. 290,996. 1928.

The process consists in limiting the degree of cracking effected so that a maximum of 25 per cent. of gasoline is produced in one pass through the plant, the stock being subjected to a preliminary heating in one or more of the fractionating stages of the system. The stock may contain light fractions, which are removed during the preliminary heating. The stock may further be mixed with, say, twice its volume of an overhead distillate —*e.g.*, gas oil or the recycle oil recovered from the reflux condenser of the plant. Crude oil may be used as the raw cracking material. H. G. S.

83. Improvements in Processes for Cracking Hydrocarbons. Gasoline Products Company Inc. E.P. 297,949.

In a process where a large reaction chamber is employed for the purpose of completing the cracking operation after the oil has been brought to conversion temperature in a heated coil as in the Cross type of plant, the converted products are withdrawn from the reaction vessel and mixed with heated raw oil at the point where pressure has been reduced but before the vapours have reached the dephlegmation stage. Cracking and vaporization are thus checked by the cooling medium and the character of the residual oil is improved, as with certain oils there is an excessive evolution of vapours, which results in a deposit of coke or asphalt difficult to eliminate from the system.

The cooling oil may be varied according to the character of the oil treated in order to produce a blend of fuel with the desired characteristics. R. P.

84. Art of Cracking Hydrocarbons. E. C. Herthel. U.S.P. 1,677,772, July, 1928.

An improved method of producing gasoline from heavy oils is claimed. The method consists in forcing the raw oil through a pipe still thereby heating it to a cracking temperature, then discharging into a chamber wherein a part of the oil is vaporized and passed through a dephlegmator.

The reflux from the dephlegmator may again be passed through the heating zone and the vapours condensed. The liquid collecting in the vaporizing drum may be discharged into a secondary drum at a lower pressure than that prevailing in the primary vaporizing drum, whereby a further separation of vapours is effected and condensed to a liquid which is passed back into the heating tube along with the fresh oil. The unvaporized oil from the secondary vaporizing drum is not returned.

In the first chamber about 35 per cent. of the original oil is withdrawn as light oil, and in the second vaporizing chamber as much as 80 per cent. may be separated.

Vaporization in this chamber may be carried far enough to leave a heavy residue or even coke. The amount vaporized may be controlled by regulation of pressure. The condensate produced from the vapours of the second vaporization is more difficult to crack than the raw oil, and may be utilised to reduce the temperature before the raw oil is charged into the system. This effect is attained by pumping the reflux through one coil of tubes, then through a second coil, and the fresh oil is supplied at a point between the coils. The furnace gases pass over the first coil, then over the second, thus ensuring the application of heat in the desired manner.

The first drum may be heated externally or may be heated internally by the hot liquid only, and the second drum is lagged with insulating material to prevent loss of heat. When vaporization is carried far enough in the second drum to produce coke, a duplicate drum may be provided in order to carry out the operation continuously. R. P.

85. Process of Converting Hydrocarbons into Lower Boiling Products. F. A. Howard. U.S.P. 1,694,262. Dec. 4, 1928.

The process consists of feeding a stream of light gas oil into a heated coil at a temperature of about 500° C. to effect a material amount of cracking in the vapour phase, then passing the products into a drum which is insulated against heat loss, along with a heavy gas oil or residue oil, and allowing the mixture to remain for a substantial period of time to permit of conversion substantially in the liquid phase.

In this manner the heating operation may be applied to clean oil, and the heat carried may aid in the conversion, under pressure, of heavy asphaltic stocks having considerable coke forming tendency. R. P.

86. Petroleum Cracking. J. J. Jakosky. U.S.P. 1,689,590. Oct. 30 1928.

Oil is conveyed under pressure through a retort at cracking temperature, the pressure being sufficient to maintain the uncracked material in the liquid phase. The cracked products are removed as vapours at a number of points distributed lengthwise in the retort. Liquid particles in the vapours are separated by electrical precipitation and returned to the retort. The apparatus claimed provides for the retort being at successively higher temperatures in the direction of oil flow. G. S.

87. Cracking Heavy Oils into Lighter Oils. E. C. R. Marks (from Petroleum Process Co., California). E.P. 299,347. June 13, 1927.

The oil to be treated is moderately heated by waste heat from the hot gases from the final exit of the apparatus, and then passed to a chamber where it is finely divided. Superheated steam is admitted to the bottom of the chamber and rises counter to the downward flowing oil. The steam and the oil vapours pass to an apparatus where additional heat is imparted. The mixed vapours are then conveyed to the bottom of another chamber to which additional heavy oil is fed at the top. The mixture of reheated gases and added oil vapour may then be passed to a third chamber and further heated, or may be condensed or passed direct to a refining system.

The preferred form of chamber comprises an outer steel shell lined with an insulating material and a second lining of a refractory and insulating material. For sub-dividing the oil stream, a series of grids are provided, each layer being in transverse relationship. G. S.

88. Cracking Stills, Controlling of. W. C. Parrish. U.S.P. 1,690,243. Nov. 6, 1928.

In cracking operations the body of oil is maintained at a cracking temperature to form light hydrocarbons and carbon, being agitated by power operated means. The preferred form of agitation is by scrapers on the inside of the chamber. The power required to operate the means of agitation is measured, and when a pre-determined value is reached, indicating an excessive production of carbon, the run is stopped. G. S.

89. Cracking Hydrocarbon Oils. J. Perl. U.S.P. (1) 1,689,362; (2), 1,689,363. Oct. 30, 1928.

A pressure tube still for the cracking of oil is arranged so that the heating gases are circulated outside the tubes. The oil under treatment is circulated through the tubes, which are coated internally with a tin-containing coating. The oil may circulate from the main supply tank, through the tubes and back to the tank, and means may be provided for the separation of suspended constituents externally of the heater and fresh oil added.

In the second patent the tubes are coated internally with an iron sulphide. G. S.

90. Process of Cracking Oil. R. T. Pollock. U.S.P. 1,688,830. Oct. 23. 1928.

The oil is heated in a coil and transferred to a vaporizing chamber, where heavy residue is drawn off and the vapours pass to a dephlegmator filled with suitable baffling material. Water and raw oil are fed into the top of the dephlegmator and flow out at the bottom into a separator. The oil layer is pumped into the heating coil and the water layer back to the dephlegmator. The light vapours from the dephlegmator overhead are condensed and collected. R. P.

91. Production of Higher Boiling Point Hydrocarbons from Low-Boiling Hydrocarbons and Hydrocarbon Gases. A. S. Ramage, U.S.P. 1,687,890. Oct. 16, 1928.

The object of the invention is to form higher boiling hydrocarbons chiefly of a saturated nature suitable as use for fuel in internal-combustion engines, or for the production of alcohols from low boiling hydrocarbons and gases such as methane, ethane, propane, etc.

The principal reactions which may be involved in the process are:—Paraffin hydrocarbons, on being brought into contact with ferric oxide at

high temperatures, are oxidised, forming unsaturated hydrocarbons. Unsaturated hydrocarbons polymerise to unsaturated hydrocarbons of higher molecular weight. Ethylene and other gases including methane react with ferric oxide forming ferrous oxide, water and carbon. Carbon reduces ferric oxide with formation of carbon dioxide at 550°/600° C. Water reacts with ferrous oxide at about 600° C., forming ferric oxide and nascent hydrogen. The unsaturated hydrocarbons are hydrogenated by the nascent hydrogen.

In the process, the proportions of hydrocarbon (oil) vapour and steam are adjusted to avoid oxidation of the ferrous oxide to ferric oxide or reduction to metallic iron.

The method employs a four inch tube 20 ft. long loosely packed with ferrous oxide. Steam and the oil vapour are supplied to this tube from any suitable source in accurately regulated quantities, one part of steam to ten parts of hydrocarbon vapour being a suitable mixture. The temperature of the tube at the outlet is preferably maintained at about 650 to 700° C., and at the inlet about 600° C.

No specific form of apparatus is claimed but a suitable type is described.

R. P.

92. Process for the Constructive Conversion of Heavy to Light Hydrocarbons. W. E. Shore. U.S.P. 1,687,595. Oct. 16, 1928.

It is the purpose of this invention to apply mechanical energy to atomise the oil in the presence of hydrogen and to produce internal heat, which is given up to the hydrocarbon. The entire mass is moved at high velocity through pipes to a compressor, where final conversion takes place.

A two cycle engine is directly connected to a reciprocating compressor, and power from the engine is transmitted to a high speed rotary pump.

Oil is pumped through the jacket of the oil engine cylinder, and the heated oil is transferred to the jacket of the compressor, absorbing the heat of the compressor. The oil is then conveyed to an expansion chamber, which is provided with a dome to facilitate the separation of volatile products. From the bottom of the expansion chamber a pipe conveys the oil through a heater, heated by the exhaust from the engine, to the rotary pump, and several conduits are connected to this pipe to convey the gases to the pump and assist in atomising the oil. The use of hydrogen or a hydrogen bearing gas for mixing with the oil is advisable, as it not only aids in atomisation, but under the temperature and velocity obtained, the minute particles are in a state of molecular instability which results in a chemical combination, as well as a physical change, gradually and progressively taking place, without the aid of a catalyst.

The speed of the reaction is controlled by the temperature, velocity, pressure, and other physical conditions present in each particular stage of the process, the most complete hydrogenation taking place in the last stage or cylinder of the compressor where the mixture is subjected to the highest temperature conditions.

The atomising pump is maintained at a uniform temperature by the circulation of mercury vapour, and gas from the pump is raised in temperature in a heat exchanger and by the hot exhaust from the engine. It is then passed through coils within a series of drums wherein the temperature is continuously raised and finally is conducted into the cylinder of the compressor. The temperature in the cylinder may range from 430° C. to 650° C. This is an essential part of the process that the heat energy of the compressor is converted into heat energy acting uniformly on the mixture, thus increasing the molecular instability of the hydrocarbons and increasing its capacity to combine with hydrogen. The highly vaporized mixture is discharged

through the series of drums to preheat the incoming mixture. The volatile vapours are conducted through a series of expansion condensers wherein different gravities of fluid are absorbed, each condenser being filled with hydrocarbon of the gravity of the oil to be absorbed in that condenser. The pressure within the various condensers is governed by expansion valves.

Uncondensed gases are passed back to the circulating pump for further use in the process. Condensate in the different drums is returned to the feed line for further conversion into lighter products. R. P.

93. An Improved Process of and Apparatus for Cracking Hydrocarbon Oil. Standard Development Co. E.P. 302,211. Dec. 12, 1928.

In specification No. 274,763 a process is described, wherein the oil after being heated to cracking temperature in a coil is passed into an enlarged digester and undergoes cracking under pressure, the cracked product being passed at reduced pressure into a still which is equipped with a fractionating tower and to which heat is supplied to vaporize a larger amount of oil than would be evaporated by the contained heat of the cracked oil.

Using a similar plant, the oil to be cracked is passed into the heated still, and the cracking coil and digester are used for cracking either a condensate from the fractionating coil or residual feed oil from the still. The contents of the digester may be discharged into the still or into the fractionation zone. A suitable apparatus is described by means of which stocks of varying grades may be treated under conditions best suited for high yields of light products. R. P.

94. Improvements in and Relating to the Conversion of Hydrocarbon Oils into Lighter Oils. Standard Development Co. E.P. 302,415. Dec. 20, 1928.

Preheated oil is pumped under a high pressure and at a temperature of below 371° C. into a heated coil in which the temperature is raised to between 399° to 482° C. The heated oil is continuously conducted into an enlarged chamber in which it is maintained at a temperature above 382° C. but below 482° C. under the same high pressure until sufficient cracking has taken place. The regulation of the temperature in the drum may be accomplished by the addition of cool oil, thus preventing the temperature of the oil within the drum from rising too high. This may be done by passing a portion of the raw feed into the drum at the point where the hot oil from the heating coil enters the enlarged chamber. R. P.

95. Process of Cracking Hydrocarbons. M. J. Trumble. U.S.P. 1,696,658. Dec. 25, 1928.

The apparatus comprises a vertical chamber with a flue, heated internally by hot furnace gases, extending centrally through the chamber. A mechanical scraper rotates about the flue, and means are provided for supplying a thin film of oil in contact with the flue within the pressure chamber. A heavy carrier oil which is stable under cracking conditions is caused to flow over the surface of the flue to remove in suspension or solution the residual products of cracking, whilst the revolving scraper mechanically prevents the deposition of the residual products on the flue, and maintains an intimate contact between the residue and the solvent oil. The vapours pass to a dephlegmator and are brought into contact with the cold carrier oil which flows from the dephlegmator to the heating surface. The vapours pass from the dephlegmator to a condenser.

The carrier oil is mixed with raw hydrocarbon and the suspended carbon partially settled. Thereafter the partially clarified mixture is returned to the cracking zone. R. P.

See also Abstract No. 17.

Oil Field Technology.

96. Apparatus for Use in Oil Wells. J. Ferguson. E.P. 299,677. April 17, 1926.

Apparatus is claimed for rapidly cutting casing when in position in a well and provides means for causing the cutter to rotate around its own axis in a path concentric to the longitudinal axis of the casing or the apparatus. At the same time the cutter is fed outwards to bite into the metal of the casing. G. S.

97. Improvements Relating to the Covering of the External Surfaces of Iron and Steel Pipes and Tubes with Bituminous Material. Stewarts and Lloyds, Ltd., and J. Mitchell. E.P. 300,933. Nov. 19, 1928.

Pipes are coated with a bituminous substance, which, by extrusion, is formed into a ribbon and wound spirally on to the pipe by the rotation of the latter. The apparatus for extruding the bituminous substance comprises a cylinder adapted to be charged with cast blocks of bitumous substance and provided with a means of heating to reduce the latter to a plastic state. The cylinder is fitted with a plunger and a shaped opening through which the plastic bituminous substance is forced on the depression of the plunger. The covered pipe may be wrapped in hessian or other material impregnated with bitumen.

In cases where it is not desirable to wind the ribbon directly on to the pipe the former may be fed on to a travelling belt and conveyed by it until firm enough to allow of its being coiled and stored. W. L.

98. Improvements in or Relating to Drills for Use in Mining, Well-drilling and Like Purposes. F. W. Thurston. E.P. 297883. Oct. 1, 1928.

The object of this invention is to provide a drill composed of separable screw-threaded sections with a form of thread which has characteristics specifically adapting it to withstand the effects of impacts from a high-duty drilling machine and also by means of which form of screw threads the parts may be readily disassembled. A rotary impact mining drill is composed of three sections—a drill rod, a bit and a coupling member. This latter is constructed with an internal screw thread and the drill rod and bit have one end of each formed with external screw threads of similar contour to engage with the screw threads in the coupling member. The screw thread, are substantially smooth, continuously curved and rounded at all parts, especially at the concave or gutter parts of the thread. Thus, in cross sections the thread has an undulatory contour, continuously curved, both the deepest and highest parts of the thread being nearly semi-circular in contour. It is preferable that these parts be made slightly less than semi-circular. In order to give an anti-fatigue property to the drill rod, wear-resisting property to the bit and shock-absorbing property to the coupling member the following heat treatment is given.

If made of Vanadium steel the drill rod is heated to 1550° F. for 30 minutes, quenched in oil and then reheated to 1100° F, for 30 to 45 minutes..

If the drill rod be made from ordinary commercial stock the first heat treatment should be at 1525° F. and the second at 550° F.

A chrome Vanadium steel bit should be heated to 1525°F. for 30 to 45 minutes, quenched in oil having a viscosity of 100 to 130 seconds at 100° F. and then reheated to 1250° F. for 45 minutes. After that lapse of time the cutting end of the bit is reheated to 1525° F. and quenched in oil, after which it is drawn or tempered at a temperature of 400° F. for a distance of approximately one inch back from the cutting edge.

The coupling member should be of chrome-molybdenum steel. It is first heated to 1550° F. for 45 minutes, then quenched in oil and then reheated to 700° F. for 45 minutes. W. L.

Petroleum Geology (Geophysics).

99. Elements of Geophysics as Applied to Explorations for Minerals Oil and Gas. McGraw Hill Book Co. Translated from "Methoden der Angewandten Geophysik," Liesegangs Wissenschaftliche Forschungsberichte Naturwissenschaftliche Reihe. R. Ambronn.

The purpose of the book is not to serve as a textbook for the execution of geophysical surveys, but first, to provide a complete and well balanced review of all the geophysical methods that may be used in economic geology so that the geologist, mining engineer, layman or executive, who has to do with them or is considering the use of them, may come as completely as possible to understand their possibilities and their applicabilities and, second, to provide for the practical geophysicists and others interested in applied geophysics, reference to the thought and literature on the subject.

The book comprises an introductory chapter on the development and present position of geophysical methods in prospecting; influence of the subsurface formations on the character of the gravitational field at the surface of the earth, including pendulum and torsion balance surveys; magnetic methods of investigations; the use of radio-active and atmospheric-electric measurements for geophysical prospecting; electrical methods of prospecting, the seismic method of prospecting, the distribution of temperature in the earth's interior and the use of temperature methods in applied geophysics.

In each subject, the elements of the pure as well as the applied theory are given, the geophysical instruments and their method of use are described, and the results of many applied geophysical surveys are shown and practically all published descriptions of geophysical surveys are mentioned. The literature on pure and applied geophysics apparently has been thoroughly searched and a detailed review is given of the pertinent thoughts, theories, suggestions, observations, data, and, at the point of mention, a direct reference is made to the authors and to the particular papers in which they have expressed an opinion on the subject.

The only adverse criticism which the reviewer has to offer is that in places insufficient distinction is made between practically untried methods which theoretically should work and methods which have actually been proved successful.

The book will be an indispensable reference work of the professional geophysicist and the textbook for elementary and advanced students of geophysics and of geologists, executives and others interested in applied geophysics. D. C. B.

100. Applied Geophysical Methods in America. D. C. Barton Economic Geology, Vol. XXII., No. 7, pp. 649–668.

The methods of applied geophysics in practical use in economic geology in America are four: (*a*) The Eötvös gravimetric method introduced by Baron Eötvös in the latter part of the past century. (*b*) The seismic method first worked out by Fessenden. Its present extensive use is largely due to an independent development of the method by Mintrop. (*c*) The electric method, which has been the subject of experimentation for a long time. The recent successful development of practical instruments and technique was due to Schlumberger, Lundberg, Nathorst and Gella. (*d*) The magnetic methods which had been in use for over half a century in connection with iron ores. Their modern extensive use in mapping geologic structure dates from Schmidt's adaptation of the Lloyd balance and Schuh's work with it on the Lübteen salt dome. The record of these methods in prospecting in the Texas Louisiana Gulf Coast has been brilliant. The present geologic status of the methods is that the instruments and technique of observation has been developed much further than the general knowledge of the geologic interpretation of the results. Research on and with the methods is being carried out by commercial companies, and the majority of the results will not be released for publication. These methods offer an interesting and almost untouched field of research both on problems of pure and applied science. A bibliography of the more important references is appended.

D. C. B.

101. The Eötvös Torsion Balance Method of Mapping Geologic Structure. D. C. Barton, Amer. Inst. Min. Met. Eng. Tech. Pub., No. 50. Feb., 1928.

An excess or deficiency of mass causes a warping of the level surfaces and of the lines of the vertical Either type of warping gives a small horizontal component to gravity at each of the two weights of an Eötvös torsion balance. That small component in one case is proportional to the differential curvature of the level surfaces, and in the other case to horizontal gradient of gravity. The formulæ for the calculation of the differential curvature and of the gradient from observations with the torsion balance are derived geometrically. The effects measured by the torsion balance are the vectorial sums of the effects produced by the topographic, the planetary, and the geologic irregularities of the distribution of mass. By running levels and using certain formulæ, corrections can be applied to compensate the effects of topography of minor relief. A correction varying with latitude is applied to compensate the planetary effects. From a study of the residual geologic anomalies, it is possible to interpret certain features of the geology. The theoretical gradient and differential curvature profiles are given for a vertical and a 30° fault cutting formations of increasing density downward, of an infinite symmetrical structural ridge, an infinite asymmetrical structural ridge, a finite asymmetrical structural ridge. Illustrations of actual torsion balance work are given as follows: the survey which led to the discovery of the Nash Salt Dome, Texas, the prediction of the limits of the dome and its verification by subsequent drilling; calculation of the extension of the cap rock on the Hoskins Mound salt dome and the subsequent verification by drilling, a profile across the Luling fault, a survey of a buried granite Ordovician ridge in Cooke County, Texas, and an analysis of a survey of the Fox oilfileds, Oklahoma. The torsion balance has a distinct place and a good future in mapping geologic structure; in some situations it works brilliantly but is not a panacea for mapping geologic structure and cannot replace but merely supplement

geology. Although it is easy to train an observer, the interpreter must be a well trained and experienced geophysicist and geologist. The torsion balance offers usefulness in certain types of pure-science geologic problems. D. C. B.

102. Modern Instruments and Methods of Seismic Prospecting, C. A. Heiland. Amer. Inst. Min. Met. Eng. Tech. Pub., 149. Oct., 1928.

For a long time it has been known that it is possible to deduce conclusions regarding the physical constitution of the interior of the earth from the records of earthquakes obtained from stationary seismographs. It is only lately, however, that practical applications of artificial explosions for geologic purposes were suggested: these culminated in Dr. Mintrop's prospecting method patented in 1919.

A very large number of portable seismographs, vibrographs, etc., now exist, and the type to be adopted for practical work depends on the completeness of the record desired. Explosives produce both longitudinal and transversal waves, the former being the faster. In practical seismic surveying only the former are measured.

In the case of a horizontal discontinuity in the subsoil there will be three types of longitudinal waves: (1) those travelling directly from the shot point to the seismograph; (2) those being refracted and travelling part of their way through the lower layer and eventually overtaking the wave through the upper layer; and (3) the reflected wave.

All three types have a horizontal and vertical component and possess a definite period, all the quantities depending on the geologic conditions beneath. If complete information is desired it is advisable to observe as many of the physical qualities of the waves as possible.

The greatest number of physical data seems to be obtainable with the mechanical seismograph, such as Mintrop's, for this type may be built for two components, has a definite individual period and magnification, and the recorded movements may easily be referred to the actual motion of the ground. The electro-magnetic seismograph is not so suitable for this type of work, because the recorded amplitude depends on the velocity of the ground displacement, not on its actual magnitude. It is often, however, an advantage to have the records of several instruments produced on one sheet of oscillograph paper.

Every type of seismograph has more or less its advantages and disadvantages, and it all depends on the purposes for which a special type is to be used.

Schweydar's two-component seismograph consists of a double pendulum and a recording device, the two parts being placed about 1 metre apart and connected with a light-proof tube.

The recorder consists of a clockwork arrangement which drives photographic paper about 6 cm. wide across a slit in front of a cylindrical lens. The light from a 3·5 volt lamp is reflected from the seismograph mirrors and falls on the paper, which is adjusted to move at from 7 to 10 cm. per sec. Time marks are obtained on the record by means of a pendulum with a pin hole, through which part of the light passes, the time interval of these marks being 0·7 secs. There is a small mirror in the recording apparatus connected to the armature of an electro-magnet; this mirror makes a line on the paper during the time that current passes through the magnet. At the moment of the explosion the current is interrupted and the line ends, thus marking on the record the moment from which the time must be counted. For this purpose the recording apparatus may be connected by a wire to the shot point, or with a radio receiver which records the moment of the explosion.

Another method, which is not very satisfactory, is to use the record made by the sound wave and compute the moment of the explosion from it.

The magnification of this seismograph is about 16,000 and could be readily increased.

For practical purposes only straight paths of the seismic waves need be considered, and the theory underlying interpretation may be handled by applying the optical laws of refraction and reflection and Fermat's principle, that the waves in passing from one point to another take the path needing the minimum of time. The question of "time travel" curves is considered at some length and references given to the original work on the subject. The theory of the curves due to faults, horizontal layers and inclined layers is given, and it is pointed out that these three cases are sufficient to interpret more complicated structures.

Methods using reflected waves are mentioned, and it is explained that they are seldom used owing to the difficulty of recognising reflected waves on a seismogram, the intensity of such waves being very slight.

Various unsolved problems in seismology are discussed, and the need for the study of certain questions explained. W. H. F.

103. The Computation of Eötvös Gravity Effects. E. Lancaster-Jones, Amer. Inst. Min. Met. Eng. Tech. Pub., 75. March, 1928.

The gravity magnitudes obtained by means of observations with the Eötvös balance in the field are necessarily resultant on total effects due to all abnormalities of mass distribution, including even the effects due to the ellipticity and rotational motion of the whole earth.

Since the object of the survey is to isolate the effects which arise solely from some particular subterranean feature, and to deduce therefrom the configuration and extent of this feature, the first step is to eliminate from the observed effects all those due to "disturbing" (*i.e.*, unwanted) features, whether above or below the surface of the ground.

The disturbing features have different characteristics, according to their situation relative to the centre of the balance and to the horizontal plane through the foot of the balance, or station point; this diversity was recognised by Eötvös, who divided the total effects into so-called Normal, Terrain, Topographical, Cartographical and Subterranean effects.

This classification has been adhered to by most subsequent investigators, but the writer prefers to employ the term "three-dimensional," which really indicates the essential character of the class of effect known as "Subterranean," at any rate from the standpoint of the computer of effects.

Before disturbing effects can be eliminated, the features must be measured up or surveyed, whether by topographical, mining, or boring methods, and then the effects of each feature calculated and subtracted from the total observed effects. After this, the problem of interpreting the residual effects will itself necessitate the computation of the assumed feature, or even a series of trial features.

The task of computing gravity effects is therefore of great importance. In the early work by Eötvös himself, the computation practically ceased when the terrain and topographical effects had been calculated, but later surveys have demanded an ever-increasing extension in the technique of computation. Certainly in surveys designed to locate minerals, particularly ores, the calculations have to be extended far beyond those of Eötvös, while his methods of computing both terrain and topographical effects have received considerable modification and improvement.

Various writers have developed isolated details of the computational programme, but the results are scattered through many technical journals,

almost always in foreign languages. Moreover, writers have generally concentrated upon the simplest types of computation and ignored the fact that actual masses which occur in Nature are apt to be decidedly irregular. The present paper is intended to act as a survey of the field of gravitational computation. Full details are given, where it is deemed necessary, of the analysis involved, and the mathematics of this has been purposely simplified even at the expense of additional elaboration. In other cases, notably the terrain computations, it is felt that the method is too well known for repetition of the analysis to be necessary.

Terrain Features.

After discussing the conditions which must be fulfilled to render standard formulæ applicable, the writer goes on to say that it is evident from the examples given that ground which slopes so imperceptibly that the average eye would not detect it can give large effects, and that the great proportion of the terrain effects arise from the ground within 30 ft. of the station. In a normal undulating region the method of computation adopted is that due to Eötvös, improved later by Schweydar.

Topographical Features.

The graticule method outlined by Nikigorov and Numerov of calculating these effects is explained, and a development of the method examined in detail, in which the centre of each sector is marked by a dot and the graticule lines subsequently rubbed out. This plan is placed on a coloured map of the survey region, and the number of dots occurring in each altitude band counted.

Three-Dimensional Features.

Formulæ 14, 15, 16 and 17 of pp. 461–2 of No. XI. of the *Journal* are given and explained, and an amplification of No. (17) added, by which the effects of an Infinite Cylinder of Polygonal Section can be calculated.

Simple quantitative rules are given to test to what extent regular features can be treated, for practical purposes, as of infinite length.

The principles underlying the calculation of effects due to irregular features are explained. The article concludes that from this review of the methods available for computing gravity effects, it is evident that this branch of gravity surveying has easily kept pace with the demands made on it, and that future requirements will no doubt be met satisfactorily.

In the field of interpretation, however, there is scope for development of a technique, and this can only be done by systematic analysis of various types of features, proceeding from the simple to the complex. Attention is being focussed upon this subject to an ever-increasing extent, and it is certain that the near future will witness a steady progress in this branch of analysis. It cannot be doubted that at present, gravity surveying is in its infancy, and that the anticipated progress in instrumental development and field technique will be accompanied by a parallel advance in analytical methods, whereby the field of application of gravitational prospecting for minerals will be vastly extended beyond its present narrow limits. W. H. F.

104. Some Applications of Potential Methods to Structural Studies. Leonardon and Kelly. Amer. Inst. Min. Met. Eng. Tech. Pub., 115. April, 1928.

The results obtained by Professor Schlumberger and his associates since 1912 demonstrate the wide possibilities of electrical exploration in structural

work. This field of activity is, at the least, as varied and valuable as the search for ore.

Ten years ago the only means of correlating the results of field work were maps of potentials with profiles and equipotential curves.

To-day the resistivity of the rocks are calculated, and the results given in the form of a resistivity plan which comprises equiresistivity curves and profiles of resistivity. These greatly facilitate geological interpretation. Examples are given of the study of concealed anticlinal and synclinal folds and faults in France, of the Boldescii anticline in Roumania and of the Meyenheim Salt Dome in Alsace. (For a more complete account of the investigations in Alsace see Abstracts 598 and 603 of Vol. 14.)

The problems discussed show that potential methods are of importance in structural work, and have passed the experimental stage. It should be noted that the method possesses great flexibility, which permits attack of a problem from several angles. This entails considerable knowledge and experience in the survey leader, and it takes more time to train a capable supervisor of electrical prospecting than of magnetometer or gravimetric work. W. H. F.

103. A New Micromagnetometer. F. Rieber. Amer. Inst. Min. Met. Eng. Tech. Pub., 119. July, 1928.

The writer refers to early magnetic instruments and modern improvements, and remarks that the limit of instrumental development is not yet reached. A word of warning is added that the ability to read smaller and smaller variations does not mean that deductions as to the causes of these variations will be made as accurately and definitely as is possible with the more pronounced anomalies, and that increased sensitivity may prove of less advantage than would be expected, due to the greater difficulty of interpretation.

The desirability of instruments with greater dependability and constancy of operation is insisted upon, and it is pointed out that all work with magnetic instruments is bound to be simplified, if it is definitely known that the readings which are being recorded are due to changes in the earth, rather than changes within the instrument.

The ordinary type of vertical magnetometer, consisting of a magnet system balanced on knife edges, is described, and its possible accuracy calculated as one part in 50,000. As the actual accuracy depends on the combined accuracy of the weighing apparatus, permanence of the magnets, forces due to circulating air currents within the instrument case, and on the leveling and stability of the stand, all of which are subject to variation, it is very much less than that calculated. The probable working error in measurements of the vertical force in places where the earth's field is 40,000 gamma is given as $\pm$ 12 gamma.

In the new device an attempt is made to overcome these inherent limitations of the usual type of magnetometer, the principle adopted being that of the vibrating-string galvanometer. In adapting this principle to magnetic measurements the usual operation is reversed, and instead of subjecting the wire to a strong magnetic field and then using it to measure weak alternating currents, a strong alternating current is passed through the wire and the movement of the wire is used to measure weak magnetic fields.

Various refinements have been introduced, the normal earth's field being balanced by a permanent magnet, etc., and it is calculated that, even with the experimental model described, an accuracy thirty times greater than that of the typical field balance can be obtained. W. H. F.

106. Use of Magnetometer in Oil Fields. L. Spraragen. *Oil and Gas J.* Sept. 27 to Nov. 15, 1928.

The instrument used in the surveys to be described was an Askania vertical field balance. It consists essentially of a moving system balanced on knife edges, this system being made up of a centrepiece, to which the knife edge is fixed, carrying a pair of magnets, mirror, lateral screws for adjusting the balance, and a vertical screw for altering the sensitivity.

The moving system, when the knife edges are lowered on to agate cylinders, comes to rest in such a position that the magnetic moments and gravity moments are in equilibrium. If the field strength is great, the angle through which the system is tilted is also great. It is thus possible to measure relative magnetic field strengths in terms of angular deflection. This deflection is measured by means of a scale carried in the eyepiece of a vertical reading telescope, reflected from the mirror on the moving system.

The mechanical refinements of the instrument consist of copper damping elements, locking device, etc. The stand of the instrument is a tripod on which is mounted a levelling head graduated in degrees with revolving table, which has four marks 90° apart.

When taking observations, the stand is adjusted above the station point, and roughly levelled by means of the circular level. The compass is then placed on the table with the guide pin in the hole provided, the moving part of the table turned until the north end of the needle reads O, and the position noted. The magnetometer is now placed on the table top with the north end of the case to the north and clamped. The revolving top is turned through 90°, the mark 2 coming opposite the reading previously occupied by 1; the instrument is accurately levelled, the moving system lowered carefully until the knife edge rests on the cylinders, and the deflection and temperature recorded. The instrument is next locked, rotated through 180° and a second reading taken. The three readings—deflection with the north end of the instrument to the east, deflection with the north end to the west and temperature—constitute the data necessary for each station.

In calibrating the instrument, two values have to be found, K the scale constant and (α) the temperature co-efficient. The scale constant is expressed as the number of gamma needed to cause a deflection of one scale division; the temperature co-efficient as so many gamma per degree centigrade.

To determine the scale constant a means of producing a field of known strength is necessary. There are two good ways of doing this, one by means of a standard magnet of known moment, the other with a large coil through which a known direct current can be passed. A suitable coil consists of two horizontal coils of 5 turns each, 48 inches in diameter, 10 inches apart connected in series and raised on a frame about 30 inches above the ground. For measuring the current a milliammeter is necessary.

For field use, a smaller coil which can be fitted directly over the magnetometer is more convenient. During surveys each instrument should be calibrated every two weeks.

The experimental determination of the temperature co-efficient requires the use of two magnetometers and a room that can be heated gradually. One instrument is set up in a basement or other place where the temperature remains practically constant, and is used to measure the diurnal variation; the other is set up in the temperature room which is gradually heated and readings of the scale deflections and temperatures taken.

In this way the part of the deflection of the instrument due to diurna variation can be deducted, giving a net result due to the change of temperature. The temperature co-efficient should be small, from 3 to 5 gamma per degree.

To obtain results for low temperatures the instrument may be artificially cooled in a refrigerator but, in this case, condensation of moisture in the instrument causes trouble.

The scale constant K should be adjusted to have a value of 20 to 30 gamma per scale division. To change the sensitivity the screw below the centre of the moving system must be screwed in or out, the small locking screw first being loosened.

To adjust the instrument for use in a particular area, the lateral screws are moved till the system balances. To extend the range of the instrument for any particular position of the lateral screws, compensating magnets are used. Three are supplied with each instrument; these are screwed firmly into the sliding holder carried on the tripod. If the magnetic moments of these magnets are known they can be used for finding the scale constant.

The intensity of the vertical magnetic force varies from place to place, the normal earth changes amounting to as much as 12 gamma per mile as one goes north and, occasionally, 3 gamma per mile as one goes west, and these variations have to be taken into account when plotting a survey.

The magnetic field also undergoes small daily changes in value, and the question of these diurnal changes, which may amount to 100 gamma in a short time, is very important in magnetic surveying. One way of measuring the diurnal variations is to maintain one instrument permanently at a base station; this is not perfectly satisfactory as it is almost impossible to correlate two continents to record the same daily variation. Another method is described later.

If the earth were made up of a homogeneous material, the magnetic field would vary very gradually from place to place. If there is a local concentration of iron ore or other magnetic material, the earth's field is distorted and the lines of force concentrated through the ore deposit, and the magnetometer readings on crossing the body show an increase. This is the principle which led to the use of the dip needle for prospecting.

In prospecting for structures associated with oil accumulations it is necessary to use an instrument more sensitive than the old type of dip needle. The problem is quite different from that of finding ore, because it is not a highly magnetic material which is being sought, but such features as granite uplifts, or other intrusions of igneous rocks, and folded sedimentary beds beneath which at some depth are basic rocks.

As a rule igneous rocks or granites have a higher magnetic susceptibility than ordinary sedimentary rocks. A granite uplift may thus be expected to cause a distortion of the earth's field in its vicinity.

In special cases the magnetometer may be used to trace lenticular sands, such as the "shoestring" sands in Kansas, for these sands frequently contain magnetite.

The magnetometer is also useful on the flanks of granite structures in tracing lenses of arkosic materials which in some areas are oil or gas bearing.

An ordinary magnetometer party consists of a chief, three operators and one or two surveyors. The chief is responsible for the supervision of the work, finance, inspection of instruments and motor equipment. He works up the field data daily. Any high school graduate with an aptitude for scientific work and experience in handling scientific instruments will make a satisfactory operator.

The cars should be light and capable of standing up under very rough service. One spare magnetometer should be carried.

There are two methods of making magnetometer surveys, the method of transverses and the method of section corners. If the section corners are

well established the second is preferable. If the region is not well surveyed readings are taken at one mile intervals along such roads as occur.

When the boundaries of a complete area for a survey are known a preliminary inspection should be made and a series of points selected as base stations; these must be far removed from pipe lines, well casings or other magnetic material. The relative magnetic forces at these stations should then be carefully measured by readings and check readings with all instruments.

The operator sets up his magnetometer at one of the base stations, and adjusts the instrument by means of one of the auxiliary magnets to read about zero. The magnet is then clamped and left in this position during the whole of the work in the area, unless the reading at any station goes off the scale when an adjustment of the magnet is necessary.

Having completed a series of readings at the base station, the magnetometer is moved to the next station. At intervals of two or three hours the operator should return to one or other of the base stations for a triple series of check readings, the last reading of the day being at a base station. The object of the check readings at base stations is to make corrections for diurnal variations and discrepancies in temperature co-efficients.

With the party described 75 stations should be completed each day.

In interpreting results the first question to consider is the susceptibilities of the formations in the survey area.

The susceptibilities of a large number of rock specimens from the Midlands of England were measured by Prof. Wilson and the results published (*v.* Royal Society Trans. A., 219, 1919, pp. 84–86). These results give a general idea of the values possessed by different types of rock. It is, however, desirable to test specimens from the actual areas surveyed, especially samples obtained from wells. A simple apparatus for roughly doing this consists of a sensitive balance with a test tube, in place of one of the pans, hanging between the poles of an electromagnet. The material to be tested is finely powdered, placed in the tube and weighed. A current producing a known flux density in the gap is then passed through the coils of the magnet and the increase of weight noted. From these data the susceptibility can be calculated.

A point to bear in mind in interpreting results is that it appears that structures are often polarized, that is, are permanently magnetized in a direction not coinciding with that of the present field of the earth.

There are three methods of interpreting results: (1) Mathematical; (2) empirical; (3) experimental.

The mathematical is only useful in the simplest cases: the empirical becomes available as soon as a large number of known structures have been mapped magnetically, while the experimental or model method is often useful.

To obtain an idea of structure, surveys over large areas are necessary, and both positive and negative anomalies are important.

In the later articles of this series detailed descriptions of magnetic measurements in Kansas and Oklahoma are given, with geological and other data. In many districts the correspondence between magnetic disturbances and geologic structure is shown to be remarkably close. W. H. F.

107. Gravity Surveying in Great Britain. H. Shaw. Amer. Inst. Min. Met. Eng. Tech. Pub., 74. March, 1928.

The suitability of the torsion balance for the location of large and important subterranean structures has given rise to a wide application of the method by many of the important oil companies.

In the search for ores a special plan of campaign, which may later be found useful in other investigations, with a close network of stations, has been found necessary, as the size of ore bodies is small compared to a salt

dome or anticline structure, and the nature of the geological and topographica irregularities is so much greater.

In cases in which the ore deposit is associated with a fault or buried river, the areas for detailed surveys may often be found with ease, but where the ore is embedded in solid rock covered with overburden the problem may prove complicated.

A general account of the employment of gravity surveying for the location of iron ore deposits in carboniferous limestone is given, together with a full discussion of the effects of subdrift topography.

As regards accuracy of results, the writer considers that the gradient values, as measured by the balance, can be relied upon to within about 1 E, while in undulating or broken ground it is difficult to make corrections accurate to 2 E.

The great drawback to the torsion balance method lies in its slowness of operation and length of time required for a detailed survey. The solution to this difficulty will probably be found in the improvement of instruments. Balances have already been designed in which the time of observation can be reduced to two hours, and the time is not far distant when a complete gradient determination at any station will be reduced to one hour.

Important developments are also likely to be introduced in methods of interpretation, by which a complete gravitational picture will be rapidly obtained from gradient measurements. W. H. F.

108. "A Background for the Application of Geomagnetics to Exploration." N. H. Stearn. Amer. Inst. Min. Met. Eng. Tech. Pub., 150. Oct., 1928.

The need for discovering new ore deposits to take the place of the known reserves, which are being so rapidly exhausted, is insisted upon, and geophysical methods are described as being of growing importance in economic geology.

Not one of the various geophysical methods is simple, though the least complicated is the magnetic.

The principle of magnetic surveying, nature of the earth's field, etc., are discussed. A list of the susceptibilities of minerals is given.

As a skeleton background for the application of magnetic surveying to geology one can visualise a deep-seated magnetic source within the earth surrounded by a magnetic field of which the ideal symmetry can be and has been established. Within this field lies the earth's crust composed of different substances grouped into rock formations. The differential permeability is definitely related to geological features, and the distortion of the earth's field can be definitely related to such features.

The geological factors which may influence the shapes of the anomalies is enormous.

The usual procedure of magnetic surveying is to run traverse lines at intervals varying from 50 ft. to 10 miles apart, and take observations at intervals on these lines varying from 12½ ft. to 2 miles, depending on the features sought.

The traverse system of surveying has the advantage of inducing a systematic survey of the area, which often brings to light geological data that might not be found otherwise. This fact has been proved by the discovery of outcrops, during the process of a magnetic survey, which would have materially altered the previous geological survey. Too much emphasis cannot be laid on the advantage of keeping a continual correlation between the magnetic and geologic data.

As regards instruments the Hotchkiss Superdip has, within the last two years, been brought to a stage of development in which it shows superiority in speed, sensitivity and directness of interpretation over the well-known Schmidt field balance.

These instruments, with the micromagnetometer described by Rieber, have obtained the greatest degree of sensitivity concomitant with practical field work.

An extensive body of empirical data is being built up by the practical application of the geomagnetic method of exploration through the use of various instruments, but since most of this work is still held as confidential, the impossibility of assembling it, for a back sight for future guidance, is obvious. Heretofore it has been impossible to predict much regarding the possible results to be expected in new applications of the method. Fortunately the method is sufficiently economical in application that it usually pays to try it out in a problematical case. An approach to a basis of prediction bids fair to lie in tabulating either the specific or relative permeability of common rocks, and to enable this to be done a simple apparatus for measuring permeabilities is desirable. W. H. F.

109. Geophysical Prospecting for Oil Resources. Anon. Oil Eng. Tech., May, 1928, 183.

The gravitational method of prospecting has now become definitely established, and only the slowness of the method and lack of portability of the apparatus has limited its application to favourable areas, such as the oil-bearing regions of Texas, Mexico, etc.

With the advent of the new design of instrument, briefly described here, the practicable disabilities of the torsion balance disappear.

The instrument, of the single beam visual reading type, is primarily intended for measuring gradients only, but can be provided with spare beams to measure curvature values or both curvature values and gradients.

When closed up for operation the instrument has a compact appearance, the lower part consisting of a tripod stand, and the upper part of a simple cylindrical aluminium case which completely encloses the balance proper.

The overall dimensions of the instrument, on its stand, are: height, 4 ft. 2 in.; diameter of outer protecting case, 7 in.; weight approximately 90 lb.

The protecting case is three-walled, of good conducting material, the separate walls being air insulated.

The suspended system consists of a circular beam carrying two equa lower masses and supporting an upper rod, on which a third mass is fixed about 45 cm. above the beam; the radial distance from the centre of the beam to each mass is 4·5 cm.

To compensate for the reduction in dimensions, as compared to the normal instruments, a specially sensitive torsion wire of co-efficient less than 0·1 is employed.

Important consequences of the reduction in the lateral dimensions of the beam system are the gain in symmetry and compactness, and the great increase in speed of operation, the beam coming to rest in about 25 minutes.

Five settings are ample in general, giving about two hours per station. Observations can be taken by day or by night. W. H. F.

110. A Magnetic Resurvey of Part of the Northamptonshire Iron Field. W. H. Fordham. *Mining Mag.*, 1928, XXXIX. (1), 18–25.

A new form of vertical magnetometer, an improved type of the Thomson-Thalen Instrument, is described, together with the results obtained with it on making a traverse survey across the bed of Northamptonshire iron ore selected in 1917 for the experimental survey carried out by the Iron Ore Committee of the Conjoint Board of Scientific Societies.

The considerations which led to the 1917 investigation, the first survey carried out to learn to what extent magnetic disturbances are connected with geologic structure, are discussed.

G. W. Walker's rule for the size of disturbing magnetic forces is given and criticised.

Walker's rule is: "The number expressing the disturbing magnetic forces is given in units 10^{-5} of an absolute unit of force, and similarly it is convenient to use a unit 10^{-5} of an absolute unit to express the susceptibility of the rocks. The rule is that the number giving the observed disturbance which we wish to explain is an approximate measure of the lowest susceptibility by which we could hope to provide a quantitative explanation. Thus if the disturbance is 100, then 100 is the order of magnitude of the minimum susceptibility which a rock must possess if it is to account for that particular disturbance."

The writer points out that this rule fails to account for the magnitude of the effects due to salt beds, and is even disproved by his own measurements made in this survey, and suggests that something of this sort might be tentatively adopted.

"The number giving the disturbance due to any rock mass is the ratio of the susceptibility of the rock to that of the formation in which it is embedded."

A copy of the field book of the survey is reproduced; this shows that observations were made at 32 stations, on a traverse of five miles, in about five hours, the magnetic disturbance being just over 300 gamma.

The writer states that the importance of Walker's work in applied geophysics has escaped notice.

The method by which the temperature co-efficient of the instrument has been reduced almost to vanishing point is explained. G. S.

111. A Gravitational Survey Over the Swynnerton Dyke, Yarnfield, Staffordshire. W. F. P. McLintock and J. Phemister. Summary of Progress of the Geological Survey for 1927. Part II. 1928.

The Calculation of Terrain Effects.

The method of evaluating terrain effects has been frequently described. A good level, an accurately divided levelling staff and a tape graduated in feet and 200 ft. long are necessary. Depending on the nature of the terrain it is necessary to take levels in 8, 16 or 32 azimuths around circles with radii of 5 ft., 10 ft., 15 ft., 20 ft., 30 ft., 50 ft., 100 ft. from the balance station. If the ground in the immediate vicinity of the station point is irregular, it may be advisable to take levels around a circle of 2½ ft. radius. The levelling must be done accurately to $^1/_{100}$ ft., particularly for the circles of smaller radius, and from the levelling data so obtained the terrain effect is evaluated by the method of Schweydar.

The following co-efficients, which have been carefully checked, were calculated for measurements made in feet from Schweydar's general formulæ. They differ materially from the co-efficients given by Fordham (*Journal*, Vol. XI., p. 456), and to a lesser extent from those given by Shaw and Jones. (In this connection see Vol. XIII., p. 148, of the *Journal*, where the re-

calculated figures due to Shaw and Jones given in italics below are to be found.)

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.
	2½ ft.	5 ft.	5 ft.	10 ft.	15 ft.	20 ft.	30 ft.	50 ft.	100 ft.	200 ft.
Uxz Uyz	26·62	22·31	35·6	9·14	2·79	1·48	·916	·435	·142	·0315
			35·8	9·15	2·81	1·47	·94	·407	·099	
Uo Uxy	23·2	38·2	49·8	27·1	13·24	10·11	9·19	7·27	4·34	1·81
			45·2	34·6	9·37	10·17	9·14	7·23	8·50	

If levelling is begun on the 5 ft. circle the co-efficients to be used are those in Columns III. to X.; if levels are taken on the 2½ ft. 5 ft., etc., circles the co-efficients to be used are those in columns I., II., IV. to X.

W. H. F.

Coal and Shale.

112. Dehydrating Tar. R. Crilly. U.S.P. 1,693,549. Nov. 27, 1928.

The tank for the reception of the tar emulsion is provided with a pipe coupling having four nipples located above the tank. The upper nipple is normally closed with a plug, the lower being fitted with a vertical pipe, the lower end of which is near the bottom of the tank. Horizontal pipes are connected to the two other nipples and a steam jet is connected to one pipe. A vertical pipe is connected to the bottom of the steam jet and projects into the tank. The plug in the upper nipple may be removed and an outlet pipe connected to draw off the tar from the bottom of the tank. G. S.

113. Improvements in and Apparatus for Use in the Treatment of Carbonaceous Matter for the Obtainment of Products Therefrom. P. Dvorkovitz. E.P. 296,793. 1928.

The apparatus is adapted for the distillation of coal and other carbonaceous matter and consists of a number of pairs of superimposed retorts. The coal is first subjected to treatment in the upper retorts, and is then allowed to fall into the lower retorts where further distillation is effected. The upper retorts are fitted with stirring gear, whilst means are also provided for the introduction of preheated coal—or producer—gas or steam into the retorts.

H. G. S.

114. Treating Aqueous Emulsions of Tar. B. Johnson. U.S.P. 1,689,728. Oct. 30, 1928.

Aqueous emulsions of tar are separated by mixing the emulsion with a carboxy acid, substantially insoluble in water but soluble in the tar, and leaving the mixture to stand at an elevated temperature until the water is separated. G. S.

See also Abstracts Nos. 76, 79, and 80.

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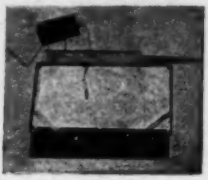

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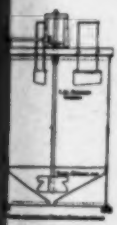

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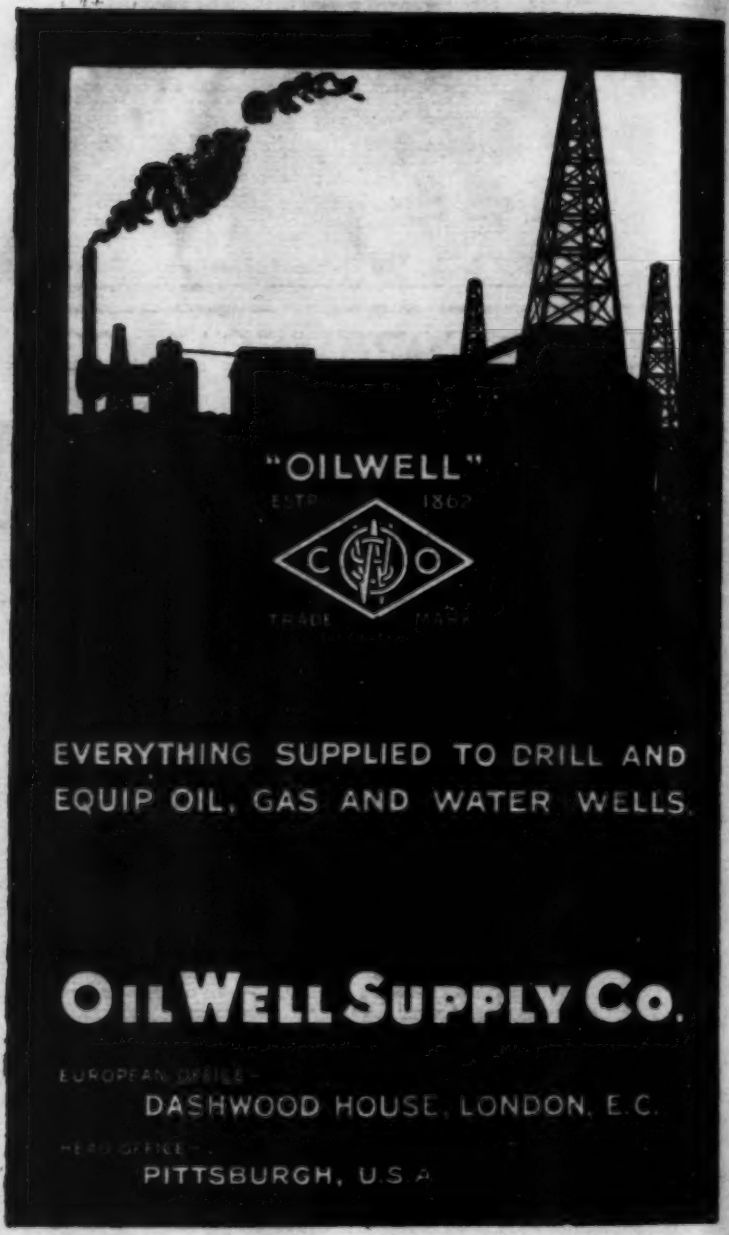
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ABSTRACTS.

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General.

115. PETROLIFEROUS BEDS OF PERSIA. C. P. Nicolesco. *Revue Pétrolifère.* 1929, 277–288.

The petroleum beds which have actually been exploited in Persia are included in the D'Arcy Concession which was first obtained on May 28, 1901, for 60 years. This area extends over 800,000 sq. kms.

Most of the petroleum is found in the northern provinces along the Persia-Iraq frontier and at the northern part of the Persian Gulf. The petroleum deposits of these two provinces are described briefly.

The northern group includes N. Persia and Afghanistan. The southern group includes Mesopotamia, S. Persia, Baluchistan and the E. Indies.

The productive petroliferous regions of Persia belong to the "petroliferous limestone provinces" which contain limestone as well as clayey and silicious rocks formed to a greater or lesser extent from cement, and limestone elements.

The mountain chains of Persia belong to the western border of the Himalaya and are arranged in two groups, northern and southern. The latter surrounds the Iranian plateau and has probably emerged from rocks of Secondary and Tertiary periods.

Certain of the structural forms in the southern group are specially characteristic—*e.g.*, those of gypsum and salt on account of their composition and mode of deposit. These rocks have become mobile under the action of tectonic forces, and the gypsum and salt have become tangentially displaced; the gypsiferous strata so formed belong partly to the upper Asmari and lower Fars groups. These arise from secondary layers which have developed further by lateral migration.

Persia occupies the fifth position amongst the world's producers of petroleum. The principal producing areas are Zokhab, Ahouaz, Naft Khana, and in particular Maidan-i-Naftun, Maidan-i-Naftek, and Maidan-i-Bibian. The best known well F7 has given, during 15 years, nearly 7,000,000 tons of petrol. This well was closed in 1926. B17 produced 1,000,000 tons of petrol per annum.

Persian crude petroleum is a brown, mobile liquid with a green fluorescence and a specific gravity of 0·837. On an average it is of mixed base and is generally accompanied by considerable quantities of natural gas, and slight traces of veteric water. The refining is carried out in the Abadan, Llandarcy, Grangemouth, Douai and Australian refineries. W. S. C.

116. NATURAL AND ARTIFICIAL PETROLEUM. A. V. Weinberg. *Petr. Zeit.* 1929, XXV. (147–151).

The first theory of the origin of petroleum was advanced by Moissan, who found that calcium carbide was formed from lime and carbon at high temperatures, and carbides of other metals were also formed. Wöhler stated further that these carbides were decomposed by water into acetylene, aluminium carbide producing methane, and these were converted by polymerisation or other chemical change into higher hydrocarbons. However, petroleum was found only to be present in the younger sedimentary layers and not in the older volcanic or crystalline formations. About 60 per cent. of the petroleum has been found in the Tertiary—*e.g.*, the petroleum deposits of Central America, California, Galicia, Roumania, Caucasus, Persia ; and abundant petroleum deposits are found in Cretaceous layers as in Mexico and Texas. Certain sand layers of the lower carboniferous as in Pennsylvania, Oklahoma, and in the great N. American region (Appalachian District) are rich in petroleum.

Engler maintained that petroleum was formed from animal remains, especially sea-animal remains. By his theory the proteins in the animal remains were first destroyed by the effect of bacteria, so that only fats were left which were gradually converted by the heat of the earth. The CO_2 simultaneously developed, usually dissolved the chalk frame or bones. Against this is the fact that no traces of such remains are found in the excessive number of petroleum occurrences, and remains of bones are only rarely discovered in oil-deposits.

In considering phenomena connected with natural petroleum occurrences, petroleum is always accompanied by an evolution of petroleum gas consisting essentially of methane, other volatile higher hydrocarbons and usually CO_2, N_2, H_2S, and H_2. In W. Virginia a pressure of 125 atmospheres, and in New York a pressure of 108 atmospheres has been recorded. The pressure necessary to produce "slime volcanoes" is over 200 atmospheres.

Another important observation is the presence of water, often salt water, usually present under the oil in the strata. This supports J. F. Carll's theory that petroleum arises from the conversion products from sea-animal remains, but this is not confirmed. H_2S, CO_2, H_2 and Helium have also been found in petroleum gas.

Regarding synthetic processes for the production of petroleum, the initial substances are lignite or coal and hydrogen. The carbon-rich coal molecules are decomposed, and the products treated with hydrogen under a pressure of 100 or more atmospheres at high temperatures and in the presence of catalysts. The CH_4 molecules are thereby converted into methanol from which benzine is obtained. The benzol content is good, and it is shown further that higher-boiling oils and paraffins remaining after the distillation of benzine can be repeatedly submitted to the same treatment.

A contact process for the production of benzine from brown coal is used in Leuna. An annual production of 200,000 tons of benzine is obtained. The optimum pressure is 100 atmospheres, and metallic oxides and salts act as weak catalysts. The effect of metallic salts present in the earth, or radioactive elements probably account to a certain extent for the occurrence of petroleum. Radioactive elements may produce 0·5 per cent. Helium as found in American petroleum.

Natural petroleum and the synthetic product exhibit optical activity, and it has been recently observed that the intensity of the optical activity in the synthetic product increases with increasing molecular weight as in the natural product. Another analogy is in the conditions of the reaction

(high temperatures and pressures), and the gaseous, liquid and solid products which have shown that water is present in both natural and synthetic petroleum. It appears therefore that the initial substances in nature and in the synthesis are the same—*i.e.*, natural petroleum arises from coal, and is of vegetable origin. It is now maintained, as stated by Potonié, that petroleum arises from coal strata. W. S. C.

Chemistry of Petroleum.

117. The Equilibrium Between Methanol, Carbon Monoxide and Hydrogen. Preliminary Paper. D. F. Smith and B. F. Branting. *J. Amer. Chem. Soc.* 1929, **51**, 129.

The equilibrium between methanol, carbon monoxide and hydrogen has been studied at a temperature of 304° and a pressure of one atmosphere. Pure methanol vapour was first passed over an active catalyst, at a rate slow enough to approximate to the ideal equilibrium conditions; by this means a maximum value for the equilibrium constant of the reaction was obtained. Secondly, a mixture of carbon monoxide and hydrogen was passed over the catalyst, thus giving a lower limit to the equilibrium constant of the reaction.

The value obtained for the equilibrium constant

$$K = [CH_3O_4]/[CO] \cdot [H_2]^2$$

of the reaction was $5{\cdot}57 \times 10^{-4}$ at 303·8°, with a probable maximum error of ±5 per cent. Using Kelley's low temperature specific heat measurements, extrapolated from room temperature, with the aid of the most accurate heat data available, a value for the equilibrium constant of the reaction was obtained, of more than one hundred times as great as the above value. Christiansen's indirect equilibrium measurements based on a Nernst approximation equation, gave a value for the equilibrium constant about 34 per cent. lower than that obtained by the authors.

The free energy equation derived from the experimental data obtained was

$$\Delta F = -20{,}857 + 41{\cdot}17\ T \log_e T - 54{\cdot}42\ T - 0{\cdot}01423\ T^2.$$

The yields of methanol as obtained practically at high pressures, agree with the value calculated from the authors' thermodynamical data. Further work is in progress with a view to eliminating errors involved in calculating the results, by constructing a very accurate gas analysis apparatus. Efforts are being made to find out why the results obtained do not agree with those calculated from Kelley's data. H. E. T.

118. Occurrence of Higher Saturated Fatty Acids in Natural Petroleum and Origin of Petroleum. Yoshio Tanaka and Tsutomu Kuwata. *Jour. Fac. Eng. Tokyo.* 1928, **16**.

Mixed naphthenic acids derived from Ishikari (Hokkaido) petroleum are shown to have a much lower specific gravity than those derived from other sources. Considerable quantities of crystalline solid acids have been isolated from the higher boiling fractions of the acid mixture. These solid acids were shown to consist of palmitic, stearic, myristic, arachidic and palmitic acids, the palmitic acid predominating. These higher saturated fatty acids have also been identified in many other Japanese, Californian and Borneo oils, but in smaller quantities than those derived from Ishikari petroleum.

The crude acids were obtained by acidifying a waste lye produced in refining gas oil distillate (29·6° Bé) of Ishikari origin, and amounted to 0·135 per cent. by volume of the distillate. The crude acid was a dark brown viscous oil having $D_{4°}^{15°}$ 0·9592, and an acid value of 94. The total yield of solid acids obtained from the crude acid was about 7·7 per cent. or approximately 0·0013 per cent. of the total gas oil distillate. The presence of saturated acids lower than myristic and higher than arachidic has been suggested.

The identification of the higher solid saturated acids in natural petroleums affords a valuable contribution to the organic theory of the origin of petroleum. The results obtained indicate that the animals responding to the formation of petroleum might have been ancient sharks and whales of the Mesozoic and Tertiary periods, since palmitic, stearic, myristic and arachidic acids have been isolated from whale oil. H. E. T.

119. Studies in Naphthenic Acids. VIII. Polymethylene Rings of Naphthenic Acids in Japanese Petroleum. IX. Some Considerations of the Chemical Constitution of Octonaphthenic Acid Derived from Nishiyama Petroleum. Tsutomu Kuwata. *Jour. Fac. Eng. Tokyo*, 1928, **17**.

Part VIII.—This communication deals with the difference in the naphthene rings present in the naphthenic acids derived from Nishiyama and Kurokawa petroleums.

The naphthenic acids were reduced to naphthene alcohols by Rupe and Langer's method (Helv. Chem. Act, 3, 272 (1920)), and then into the corresponding naphthene iodides, being finally reduced to naphthene hydrocarbons by means of zinc amalgam.

In order to ascertain the presence of hexamethylene rings in the naphthenic acids, the naphthene hydrocarbons were treated with platinum black at 300° C, the extent of decomposition being determined from the volume of hydrogen evolved and the change in specific gravity and refractive index of the hydrocarbon. Homologues of hexamethylene carboxylic acid are thus shown to be present in small amount in Kurokawa, but not in Nishiyama naphthenic acids.

Part IX.—The chemical constitution of octonaphthenic acid (heptanaphthene-carboxylic acid) derived from Nishiyama petroleum has been studied.

A monobrominated derivature was produced by the application of the Hell-Volhard-Zelinsky method. The α-monobromo-octonaphthenic acid thus formed was treated with either aqueous or alcoholic potassium hydroxide, the corresponding α-hydroxy acid being thus produced. The α-monohydroxy-octonaphthenic acid was then oxidised by means of lead peroxide, the products of the reaction being carbon dioxide and a neutral oil containing a small quantity of an aldehyde. This neutral oil gave a considerable quantity of iodoform with iodine and potassium hydroxide, and when decomposed with alkaline potassium permanganate it yielded a large quantity of neutral crystals and a small quantity of solid acid.

A further study of these compounds is being made, but from the results obtained the chemical constitution of octonaphthenic acid was supposed to be the homologue of methyl-pentamethylene carboxylic acid, having no tertiary carbon atom in the α position to the carboxylic group. H. E. T.

120. THE DESULPHURISING EFFECT OF SILICA GEL. III. By H. Waterman and M. Tussenbroek. *Brennstoff-Chem.* 1928, **9**, 397-398.

In the first two articles in this series the desulphurising effect of silica gel on aliphatic sulphides, disulphides and mercaptans was discussed, and it was found that the effect was of the same order of magnitude for all those compounds which the authors investigated.

The present paper deals with the action of silica gel on aromatic sulphur derivatives and thiophen, also on colloidal and dissolved sulphur. Thiophens, diphenylsulphide, dibenzyl sulphide and benzyl mercaptan were used.

The colloidal solution of sulphur was prepared by Garard and Colt's method. H_2S prepared from FeS and HEl, and SO_2 from $NaHSO_3$ and HEl were washed, dried with $CaEl_2$ and passed into 200 gms. kerosine. The sulphur separated gradually, was removed by filtration and the filtrate treated with a stream of pure nitrogen to remove excess SO_2 and H_2S. The solution obtained was stable at high temperatures, and could be concentrated without a separation of sulphur occurring. A liquid containing 0·126 per cent. sulphur was obtained which was finally concentrated in a vacuum to about $\frac{1}{3}$ of its original volume.

Silica-gel was applied as in the previous researches. Tables are given showing the size of the grain and the moisture content of the gel. Solutions of diphenyl sulphide, dibenzyl sulphide and benzyl mercaptan in kerosene were utilised, and results showed that adsorption took place to a considerable extent and apparently to a greater extent than in the case of the aliphatic compounds.

The decrease in sulphur content caused by the adsorption effect of silica gel is shown by the following :—

Quantity of Silica Gel applied to 50 ccs. Solution.	Sulphur Compound.	S content. Before Silica Gel Treatment.	S content. After Silica Gel Treatment.	Temperature.	Time of Shaking.
4 gms.	S	0·26	0·24	Room temp.	1 hr.
,,	$(CH_3)_2CH.CH_2CH_2SH$	0·29	0·09	,,	,,
,,	H_2S	0·034	0·03	,,	,,
,,	$(CH_3)_2SO_4$	0·04	0·00	,,	,,
,,	$p.CH_3.C_6H_4.SO_3CH_3$	0·15	0·03	,,	,,
,,	CS_2	0·08	0·08	,,	,,
,,	$(CH_3.CH_2.CH_2.CH_2)_2S$	0·30	0·13	,,	,,
,,	$(CH_3.CH_2.CH_2)_2S$	0·38	0·24	,,	,,
,,	C_4H_4S	0·09	0·08	,,	,,
,,	$(C_6H_5)_2SO$	0·05	0·00	,,	,,
,,	$(CH_3CH_2.CH_2)_2SO_3$	0·08	0·00	,,	,,

Wood, Sheely and Trusty used a solution of thiophen in benzene which contained 0·09 per cent. sulphur. The sulphur content after treatment with silica gel was 0·08 per cent.; the gel also had no effect on H_2S, CS_2 and free sulphur. Sulphur is neither absorbed from colloidal solution nor from molecular solution by silica gel.

Finally the effect of silica gel on kerosene itself has been investigated. As criteria for the resultant effect, the change in aniline point was utilised. The kerosene in question showed, before refining with concentrated sulphuric acid, an aniline point of 53·3°, and 71·2° after refining with concentrated

sulphuric acid—*i.e.*, an aromatic content of 21 per cent. The unrefined kerosene was mixed with 20 per cent. silica gel and shaken at 31·31° for six hours. The aniline point was found to increase with all sizes of grain. An increase of the aniline point of 1·1°–1·2° shows that the adsorbing effect of the silica gel extends preferably to the aromatic constituents. W. S. C.

121. New Synthetic Resins from Aromatic Hydrocarbons. P. Krishnamurthy. E.P. 298,939. Jan. 24, 1929.

Resins are produced by the action at ordinary or slightly elevated temperatures of anhydrous zirconium tetrachloride or anhydrous ferric chloride on aromatic hydrocarbons mono-chlorinated in the side-chain, the process being conducted in a suitable solvent. Thus, benzyl chloride in about an equal quantity of carbon disulphide is treated with about 1–2 per cent. of zirconium tetrachloride, the solvent removed and the product freed from soluble zirconium salts by the action of water and steam, and the yellow solid resin extracted by means of hot benzene or coal-tar naphtha. After distilling off the solvent, the residue is melted, heated and stirred at about 200°, yielding a transparent, amber-coloured, brittle solid, inert to acid, alkali or salt solution, which may be blended with rubber, etc., to produce insulating material. W. S. N.

122. Improvements in the Manufacture and Production of Diolefines. J. Y. Johnson. E.P. 303,323. Jan. 3, 1929.

A process is described for the recovery of butadiene or similar diolefines from gaseous mixtures. The process consists essentially of treating the gaseous mixture containing the diolefines with a salt of a heavy metal such as silver or mercury, occurring in Groups I. and II. of the Periodic System. By this means an additive compound of the salt with the diolefine is formed, from which the diolefine can be recovered in a state of purity by warming or by treatment with hydrochloric acid. H. E. T.

123. Manufacture and Production of Olefines and Diolefines. J. T. Johnson. E.P. 303,998. Jan. 17, 1929.

Olefines and diolefines are produced by bringing the vapour of paraffins, cycloparaffins, olefines or cyclo-olefines or mixtures thereof into contact with a catalyst containing or consisting of lustrous carbon, prepared as described in Ber., **56**, 2071; **59**, 2433. The temperatures employed are preferably 500–800°, more particularly 550–750°. It is advantageous to employ carbon deposited on carriers, particularly the difficultly reducible metal oxides or compounds—*e.g.*, beryllium oxide, tertiary calcium phosphate, also aluminium oxide, chromium oxide, active silica, natural or artificial silicates, quartz or porcelain, or on metals such as copper or chromium. When operating in the presence of water vapour it is inadvisable to employ such oxides or compounds (*e.g.*, alkali or barium oxide, or mixtures of barium oxide and calcium oxide) as accelerate the water-gas reaction. The process is preferably conducted at ordinary or reduced pressure, and preferably in the presence of diluents. Water vapour is particularly useful for this purpose; the use of lustrous carbon does not lead to the formation of appreciable amounts of carbon oxides. Examples are given of the treatment of *cyclo*-nexane, an American petroleum and a synthetic benzene from the destructive hydrogenation of brown coal. W. S. N.

See also Abstracts Nos. 130, 152, 153, and 253.

Crude Oil.

124. Refining of Gasoline Distillates from West Texas Crudes. H. H. Hewetson. American Petroleum Institute Bulletin, 1929, **10**, No. 2, 79–82.

Crude oils from Crane-Upton, Winkler and Pecos, which are in general representative of West Texas crudes, have gravity 28°, 29° and 30° A.P.I., 2 per cent., 1·5—1·75 per cent. and 1·25—1·75 per cent. of sulphur, 20 per cent., 20–25 per cent. and 20–25 per cent. of gasoline, and 0·16 per cent., 0·20–0·23 per cent. and 0·18–0·20 per cent. of sulphur in 410° F. end-point, untreated gasoline, respectively. On distillation to 600° F. (vapour) 1–2 gm. of hydrogen sulphide is evolved per litre of any of these crudes. The overhead fractions are extremely difficult to sweeten. Various refining schemes have been employed for dealing with West Texas oils. If the crude is topped to give a straight-run gasoline of good end-point and sulphur content 0·16–0·25 per cent., and this gasoline is acid-treated and sweetened with plumbite without re-running, a finished gasoline containing 0·10–0·15 per cent. sulphur, but of somewhat high gum content, will be obtained. If, however, the acid-treated stock is re-run with the addition of gas oil or sweet crude bottoms, the finished gasoline will have a good gum test and sulphur content below 0·10 per cent. Frequently a distillate is produced containing 80–90 per cent. of end-point gasoline, which requires 10–15 lb. of 66° Bé. acid, and when re-run and sweetened gives a gasoline of sulphur content below 0·10 per cent. and having a fair gum test. The cracked gasoline from either a residue or a heavy distillate is much more difficult to refine than the straight-run gasoline. Probably the simplest procedure would be to run in addition enough sweet crude to give a satisfactory blended cracked gasoline. As a last resort the distillate may be twice acid-treated and twice re-run. Acid as dilute as 66° Bé. is too weak for use on West Texas unrefined cracked distillates. Acid stronger than 98 per cent. does not give greater sulphur removal than does a larger quantity of either 98 per cent. or 66° Bé. acid and removes an unduly high proportion of desirable unsaturated constitutents and gives a gasoline unstable to light and of high gum content. 98 per cent. acid is more effective than 66° acid, but the stronger grade leaves compounds in the distillate (even after a soda-wash) which evolve free acid on re-running. Although a large excess of acid (e.g., 30 lb. 66° acid per bbl.) will give a large reduction in sulphur content (0·38 per cent. to 0·03 per cent.) the treating loss and costs are high. With an increase in temperature of treatment, less acid of a given concentration or weaker acid in a given quantity will effect the same result. In treating West Texas distillates, not only the time of agitation, but also the time of settling should be reduced to a minimum, since the resinifying action of the acid continues after the solvent action on sulphur compounds has ceased. A preliminary treatment with plumbite or hypochlorite tends to reduce the sulphur in the treated distillate, but the same result is attained less expensively by using more acid; the same is true of a plumbite or hypochlorite treatment before re-running. With West Texas stocks an initial soda-wash is of limited value. If, after acid treatment, the distillate (0·28 per cent. sulphur) is neutralised with clay, the sulphur content is less (0·15—0·18 per cent.) than if neutralised with soda (0·26 per cent.), but on re-running, the sulphur content of the distillate is the same (0·12 per cent.). Sweetening, but not appreciable desulphurisation, is accomplished under certain conditions by vapour-phase treatment; fuller's earth, bauxite, pumice wet with phosphoric acid, and calcium, barium, cadmium and aluminium oxides are employed. Aqueous copper chlorides do

not affect the distillates and mercuric chloride forms petroleum-soluble, organic mercury derivatives. The use of saturated aqueous mercuric acetate followed by acid reduces the sulphur content more than acid alone. Anhydrous chlorides of trivalent and tetravalent metals, particularly aluminium, antimony and tin, sweeten the gasoline but do not desulphurise it. Aluminium chloride has a very marked action in polymerising the unsaturated constituents. W. S. N.

125. Process of Treating Petroleum Oils. J. C. Black. U.S.P. 1,704,588. March 5, 1929.

Crude oil containing emulsified water and corrosive salts is mixed with an alkali water solution, and the mixture heated to complete the reaction of conversion of the corrosion salts to non-corrosive compounds. The major portion of the water and non-corrosive compounds is separated under pressure, and the heated oil mixed with hotter residual oils to complete the separation of the water. G. S.

126. Method of Treating Oils for Distillation. J. M. McClave. U.S.P. 1,703,158, Feb. 26, 1929.

A method is described for treating oils for the purpose of promoting the efficiency of distillation and to obtain a higher percentage of low boiling hydrocarbons by the decomposition of all or part of the high boiling hydrocarbons.

Manganese dioxide is first introduced into the oil in proportion to the percentage of unsaturateds present, after which the oil is agitated and the proper quantity of hydrochloric acid added. The free carbon is thus changed from a colloidal to a flocculent condition. The sludge which separates out is then easily removed. The oil is now ready for distillation, and it is advantageous to add the same quantities of pyrolusite and hydrochloric acid at the time of distillation for the purpose of obtaining a high percentage of low boiling oils. The percentage of reagents used will vary with the nature of the oil under treatment. H. E. T.

127. Method of Refining Petroleum Oils. O. Dieckmann. U.S.P. 1,703,615, Feb. 26, 1929.

Crude oil is topped to remove the lighter fractions and the heavier oils and asphaltum compounds are washed with sulphuric acid. The acid sludge separated is washed with refined petroleum oil to recover the acidulated oil in the sludge. The washing oil and recovered oil are returned to the main body of acidulated oil, which is then neutralised and fractionated. G. S.

128. Process of and Mixture for Cleaning Crude Oil. W. L. Palmer. U.S.P. 1,699,374, Jan. 15, 1929.

The separation of oil from water and other foreign material is accelerated by the addition of a mixture consisting of approximately one part oxalic acid dissolved in one part glycerin, two parts glacial-acetic acid, 48 parts cresylic acid and 48 parts oleic acid, used in the proportion of one gallon of the mixture to one hundred barrels of crude oil. Gravity separation takes place on standing from 2–24 hours. H. S. G.

129. Method of Cleaning Crude Oil. E. A. Sperry. U.S.P. 1,699,379, 1929.

Crude or bunker oil is refined by centrifuging and heating the oil during its passage through the machine. In cases where there is liability for vapour to form the process is carried out under pressure. R. S.

See also Abstracts Nos. 118, 119, 245, and 247.

Natural Gas.

130. The Washing of Hydrogen Sulphide from Industrial Gases by Means of Alkaline Potassium Ferricyanide Solutions. Fischer and P. Dilthey. *Brennstoff-Chem.*, 1928, **8**, 122.

This paper deals with the effect of potassium ferricyanide in removing H_2S according to the equation :—

$$2K_3Fe(CN)_6 + H_2S + 2KOH = 2K_4Fe(CN)_6 + S + 2H_2O$$

and the possibilities of the regeneration of same.

A gas containing H_2S was produced by passing a stream of pure hydrogen through an absorption flask filled with a 50 per cent. saturated solution of H_2S and water rising to a height of 15cms. The velocity of reaction of alkaline $K_3Fe(CN)_6$ with H_2S was very great even at room temperature. Sulphur separated in a yellow crusty form, which had a melting point of 116–117°, and a small portion of the sulphur was conveyed with the gas in the form of vapour (estimated at about 0·1 per cent). There are two volumetric methods for the control of the reduction of $K_3Fe(CN)_6$, namely : (1) $K_4Fe(CN)_6$ which is formed is determined by oxidation with $KMnO_4$ solution ; (2) total iron content after reduction is determined, also " ferri " content before and after experiment determined iodometrically, the difference of both values giving the ferro-content.

The oxidation method was used for the majority of the experiments.

Research on the Influence of Alkalies.—The two factors which influence the washing process are the concentration of the alkali and of the $K_3Fe(CN)_6$. The influence of these extends to the sulphur yield on the one side, and the velocity of the reaction on the other side. A distinction is made between the S yield as present in the gas and the S obtained by weighing and the consumption of $K_3Fe(CN)_6$ calculated from the weighed S and the iron titration. The consumption of $K_3Fe(CN)_6$ in S formation is more important here, especially with regard to regeneration costs. The S yield first mentioned is interesting from the point of view of secondary reactions. The H_2S content was found by two methods : (*a*) H_2S was displaced by H_2 with a definite streaming velocity, the H_2S content determined by passing a definite quantity of gas through an intermediate I_2 solution and I_2 determined by back titration. The mean value of this S content/unit volume of both titrations multiplied by the quantity of gas in the principal experiment gives the required quantity of sulphur. (*b*) S filtered through a Gooch crucible, dried and weighed. The results of (*b*) were uncertain. It was found better to utilise a greater quantity of $K_3Fe(CN)_6$ for the principal experiment (500 ccs.).

The significance of the consumption determined by weighing the sulphur directly and estimating the iron by titration is shown by the following equations :—

(1) $4K_3Fe(CN)_6 + 2S + 6KOH = 4K_4Fe(CN)_6 + K_2S_2O_3 + 3H_2O$.
(2) $4K_3Fe(CN)_6 + S + 6KOH = 4K_4Fe(CN)_6 + K_2SO_3 + 3H_2O$.
(3) $6K_3Fe(CN)_6 + S + 8KOH = 6K_4Fe(CN)_6 + K_2SO_4 + 4H_2O$.

Thus 1 gm. atom of sulphur requires 2 molecules $K_3Fe(CN)_6$ in (1), 4 molecules $K_3Fe(CN)_6$ in (2), and 6 molecules $K_3Fe(CN)_6$ in (3) for neutralisation—*i.e.*, four times the quantity of $K_3Fe(CN)_6$ is required in order to oxidise H_2S to sulphate instead of sulphur. A further oxidation of sulphur must therefore be avoided. The results of the sulphur yield with different alkali additions to 60 ccs. at a time of 15 per cent. $K_3Fe(CN)_6$ solution are shown numerically in tables. The consumption is found to increase with

decrease of alkali concentration. Glund and Schönfelder tried the effect of soda instead of alkali, ½ molecule soda added per molecule $K_3Fe(CN)6$. The velocities of consumption and washing are found to be inversely proportional to one another. From an economic point of view for the production of sulphur only it is best to use $K_3Fe(CN)_6$ with lower alkalinity. The velocity of reaction and the sulphur yield are improved by adding bicarbonate to the carbonate.

The influence of the $K_3Fe(CN)_6$ concentration on the washing process is next considered. The solution used contained 1 $K_3Fe(CN)_6$/1 Na_2CO_3, with the addition of 10 per cent. $NaHCO_3$. The concentration of $K_3Fe(CN)_6$ is found to have no influence on the consumption, but it has a great influence on the velocity of reaction.

Experiments on the Degree of Reduction of $K_3Fe(CN)_6$ Solution with H_2S Present in the Final Gas.—It is essential to determine the relation $K_4Fe(CN)_6$/$K_3Fe(CN)_6$ which is to be maintained in a continuous washing process in order to remove H_2S quantitatively from the gas. With strongly alkaline solutions, all $K_3Fe(CN)_6$ is reducible to $K_4Fe(CN)_6$ solution before H_2S occurs in the end gas, whilst concentration with lower alkalinity is of small influence in this respect. This may be explained by the occurrence of a definite equilibrium in the formation of carbonate from the added bicarbonate; this is brought about by an intermediate sulphide formation which takes place slowly owing to the approximately equal acidities of H_2S and CO_2. An increased alkali addition is necessary for further consumption of the solution. The effect of the addition of sodium bicarbonate is found to be very favourable.

Experiments to Explain Secondary Reactions.—The sulphur produced from H_2S was shaken with fresh $K_3Fe(CN)_6$ solution in a closed Erlenmeyer flask and allowed to stand. From time to time a sample of the solution was removed and its ferro-content determined by titration. This increased very slowly even with strongly alkaline solutions. A sulphur when treated with a 15 per cent. $K_3Fe(CN)_6$ solution containing 5 KOH/1 $K_3Fe(CN)_6$ showed that the percentage of ferro-content increased with time. Sulphur when treated with a saturated $K_3Fe(CN)_6$ solution containing 1 Na_2CO_3/-1 $K_3Fe(CN)_6$ also showed an increased per cent. ferro-content with time. The blowing of air through a mixture of S and $K_3Fe(CN)_6$ solution at 50–70° has only a very slight effect. Secondary reactions appear to be caused principally by the formation of sulphuric and thio acids.

Discussion of Various Possibilities of Regeneration of $K_3Fe(CN)_6$.—Gluud and Schönfelder attempted to regenerate $K_4Fe(CN)_6$ with chlorine, and with PbO_2 and other oxidising agents in the presence of CO_2, also the conversion of $K_4Fe(CN)_6$ into $K_3Fe(CN)_6$ at increased temperature and pressure.

A quantitative conversion was impossible owing to decomposition. The authors have tried the effect of ozone:—

$2\ K_4\ Fe(CN)_6 + O_3 + H_2O = 2\ K_3\ Fe(CN)_6 + 2\ KOH + O_2$, thus 32 gms. $S = 2\ K_4Fe(CN)_6 = 48$ gms. O_3; also regeneration by electrolysis but have obtained no satisfactory results. W. S. E. C.

131. Purifying Gases from Organically Combined Sulphur. F. Fischer. E.P. 282,634. Jan. 24, 1929.

Gases are purified from organically combined sulphur by passage at 200–300° over contact substances containing precious metals of the first group of the periodic system, especially silver, in the presence of hydrogen, the hydrogen formed being then removed in the usual manner. The temperature necessary is lower than when lead or antimony is employed, and the composition of the gas is not otherwise modified as when iron, nickel or cobalt is used, W. S. N.

132. Transformation of Gaseous Hydrocarbons. P. H. Hull and Imperial Chemical Industries, Ltd. E.P. 304,914. Jan. 21, 1928.

Gaseous hydrocarbons are treated by electrical discharges to effect chemical reactions by passing them through an arc between poles of tungsten, tungsten carbide, molybdenum, osmium or tantalum. G. S.

133. Apparatus for Combining Natural Gas and Hydrocarbon Oil for the Production of Gasoline. L. S. Worthington, assr. to C. L. Thompson. U.S.P. 1,700,556. Jan. 29, 1929.

Oil is fed from a supply tank to a feeding tank in which it is maintained at a predetermined level. From the feeding tank the oil passes through a heated coil to an atomiser inlet in the feeding tank, which is also the treating tank. Heated gas is also fed to a jet in the atomiser, and means are provided for conveying the vapours formed at the inlet to a suitable condenser. G. S.

See also Abstract No. 263.

Motor Spirit.

134. Importance and Significance of Gum in Gasoline. V. Vorhees and J. O. Eisenger. American Petroleum Institute Bulletin, 1929, **10**, No. 2, 169-180.

Since much of the gum formed when gasoline is exposed to sunlight is soluble, this test for gum stability is of little value; moreover, a gasoline may be satisfactorily stable in the dark but may deposit gum in the light.

The commonly used copper dish test does not discriminate between gum already present and that formed during the test. The possible catalytic effect of copper is not met with in actual storage. There is lack of oxidation control. Moreover, volatile fractions are exposed to atmospheric oxidation for a shorter time than heavier constituents, hence the test, even if it could be standardised (*e.g.*, as regards rate of evaporation), would give results varying with the boiling range of the fuel.

Preformed gum (*i.e.*, gum already present) is estimated by evaporating 50 c.c. samples of gasoline to constant weight (this requires about 10 hours) in 200 c.c. Pyrex dishes supported at three points (to avoid mechanical loss of resin, which tends to creep) on the shelf of an oven, maintained by external heating at 320–350° F., into the bottom of which steam is introduced.

Gum formation is studied by keeping samples of gasoline at 210° F. in an enclosed apparatus filled with oxygen initially at about 2.5 atm., the absorption of oxygen being followed by means of a manometer. There is always a distinct period of induction during which slow but uniform oxidation occurs; this is followed by a period of much more rapid oxygen absorption. The differences between gasolines seem to be characterised chiefly by differences in the induction periods, after which all gasolines oxidise to form gum at about the same rate. Within reasonable limits, the amount of gum formed seems to depend only upon the oxygen available and the time of heating. There is fair proportionality between the oxygen absorbed and the weight of gum produced, measured by the steam oven test.

For routine determinations of gum stability, the oxygen gum test is used. The sample of gasoline (25 c.c.) is introduced into a 500 c.c. round-bottomed, long-necked Pyrex flask previously filled with oxygen. The stopper is tied in and the flask immersed to the neck for exactly five hours in an actively boiling steam bath. After cooling, the contents are transferred and washed

(using benzene-alcohol) into a dish and evaporated as in the steam oven test. Stable gasolines give 2–10 mgm. (8–40 mgm. per 100 c.c.), whereas unstable ones give as much as 500 mgm.

Figures are given to show that the oxygen gum test gives more consistent results than the copper dish test, and to show the entire lack of correlation between colour and gum content in a gasoline.

Comparative tests are made in which five gallons of each fuel are consumed in a single-cylinder air-cooled Delco engine, model 850, and the gum deposited in the intake system (inlet passage and inlet valve) is measured. In general there is an evident correlation between the gum deposit and the preformed gum content of the fuel as determined by the steam oven test. The gasoline is not in the intake manifold sufficiently long for oxidation and gum formation to occur, and only gum which has previously been formed is deposited on the hot surfaces. The results, admittedly incomplete, seem to indicate that more than 10 mgm. of actual or preformed gum per 100 c.c. of gasoline at the time of use may produce a noticeable deposit of resin in the inlet passages and on the valves.

Figures are given which indicate that a gasoline which forms less than 10 mgm. of gum per 25 c.c. in the oxygen gum test will not form enough gum to be impaired in value on storage for at least a year. W. S. N.

135. Rectification of Gasoline Fractions. H. D. Wilde, B. Mead and S. P. Coleman. *Oil and Gas J.*, 1929, **42**, 102.

The work recorded in this paper has to do essentially with the determination of vapour pressures below atmospheric pressure. An attempt is made to correlate the results obtained with their practical value in refinery operations.

The method consists in bringing a known quantity of air into equilibrium with the gasoline whose vapour pressure is to be determined and measuring the increase in volume produced. Under the conditions of equilibrium at the temperature of the experiment, the proportion of hydrocarbon vapour in the air-vapour mixture will be in the same proportion as the vapour pressure of the gasoline is to the total pressure existing. Thus by Dalton's law, the increase in volume of the air over the total volume, multiplied by the total absolute pressure existing, will be the absolute vapour pressure of the sample under test.

The apparatus consists essentially of a gas burette and a saturator each immersed in a water bath. The following equation is employed for calculating vapour pressures :—

$$\text{Vapour Pressure} = \frac{\text{increase in volume}}{(\text{vol. of air} + \text{inc. in vol.}) \times \text{barometer reading.}}$$

Vapour pressure measurements afford a means of controlling refining operations in order to determine what should be included in the gasoline fraction of a crude and what should be excluded. Further work now being carried out has shown a close correlation between vapour pressures as determined by the method described in this paper and those calculated from Raoult's law from the known chemical composition of light gasoline fractions. With methods available for analysis of gases and light gasoline in terms of pure hydrocarbons, it is thus possible to survey plant operations and determine what recoveries may be effected on the basis of proper vapour-pressure control. Field and plant tests have also shown a close relation between true vapour pressure and evaporation losses. Further work indicates that vapour pressure may yield a fair amount of information on the performance of gasolines under the conditions of use in internal combustion engines.

H. E. T.

136. An Improved Process and Apparatus for Treating Hydrocarbon Oil. Imperial Oil Limited. E.P. 305,108. Jan. 30, 1929.

The process consists of bringing the vapours of naphtha or gasoline into contact with a counter-current stream of liquid carrying finely-divided clay and maintaining the temperature sufficiently to cause the desired product to leave the treating zone as vapour. The apparatus comprises a tower having a vapour outlet at top, means for supplying a stream of liquid containing the clay to the tower below the vapour outlet, vapour liquid contacting means with the tower and an oil vapour line discharging into the bottom of the tower. The oil is fed to the bottom of a column through the vapour line into a shell still wherein it is volatilized. The column may be of the usual bubble-cap type with a cooling coil in the upper part. The vapours from the column pass to the bottom of the treating tower or the column may be used as a combined dephlegmator and treating tower. There is a settling tank connected beneath the treating tower to receive the solid absorbent and reflux. The reflux is pumped back into the fractionating column and a bottom draw-off is provided to remove the absorbent. It is preferable to use an oil which has been through the process as a carrier for the finely-divided clay, and a steady supply of the mixture is pumped into the tower below the topmost plate.

The amount of clay required will vary with the composition of the raw oil, but in general 2 to 5 per cent. of the weight of stock is sufficient. The clay treatment takes the place of acid treatment and re-running of cracked distillate. The distillate obtained overhead is of good colour and stability.

Metallic oxides may be supplied along with the clay in cases where the oil contains a high percentage of sulphur compounds. R. P.

137. Process and Product for Purifying Petrol. W. M. Knowling and M. Kostevitch. E.P. 287,141. Feb. 7, 1929.

This invention relates to a method of refining petrol, whereby impurities such as water, tar, pitch, metallic salts and sulphur compounds are removed. The refining agent consists of a mixture of activated silica gel and activated charcoal.

An attempt is made to explain the theory of the refining action of the mixture of charcoal and silica gel. H. E. T.

138. Preparing Hydrocarbon Products. N. E. Loomis, Assr. to Standard Oil Development Co. U.S.P. 1,694,272. Dec. 4, 1928.

The invention claims an improvement in the art of preparing hydrocarbon products, and comprises introducing a light condensate containing some hydrocarbons which are normally gaseous into the intermediate region of an absorber tower. The vaporising constituents are passed up through progressively cooler zones at a pressure of 100–300 lbs. per square inch. A flow of heavy naphtha is supplied counter-currently, and a substantially balanced gasoline is drawn off from the bottom of the tower. S. W. C.

139. Purification of Benzol, Petrol and the Like. K. Cox and P. J. McDermott. E.P. 303,991. Jan. 17, 1929.

This an improvement on the process of E.P. 269,242. Hydrocarbon oils are refined by refluxing with ferric sulphate, filtering, neutralising with alkali and redistilling. W. S. N.

See also Abstracts Nos. 124, 140, 182, 196, 216, 218, and 239.

Kerosine.

140. Refining Hydrocarbon Oils. H. J. Broderson. U.S.P. 1,698,428. Jan. 8, 1928.

This invention relates to improvements in refining hydrocarbon oils such as naphthas, pressure distillates and burning oils.

The oil is pumped from a source of supply through a preheater, where its temperature is raised to between 80° and 100° F. From the preheater, the oil passes to a mixer in which it is thoroughly mixed with the treating liquid. From the mixer, the oil containing admixed treating solution is passed to a settling tank, where the oil and treating solution separate. The treated oil continuously overflows from a pipe at the top of the settling tank, while the separated treating mixture is withdrawn from the bottom and pumped back into the mixing tank in the path of the incoming oil.

The treating solution may be "doctor" or sodium plumbite solution as ordinarily used in refinery practice. H. E. T.

See also Abstracts Nos. 150, 155, and 196.

Fuel Oil.

141. Method of Preparing Liquid Fuels for Combustion. Dehn and the Gasone Corporation. E.P. 286,386. March 8, 1928.

The fuel mixed with air and water is subjected to emulsifying action by means of three low pressure pumps, after which it is subjected to further emulsification under high pressure and immediately released to atmospheric pressure into the combustion chamber. Thus, the mixture is delivered into the combustion chamber with explosive violence permitting high disintegration of the fuel. A. R. S.

142. Treatment of Oil Fuel Refuse as Obtained After Clearing and Washing Out of Tanks or Bunkers. H. W. Wildish. E.P. 301,405. July 28, 1927.

The oil residue from the cleaning of ships' fuel oil tanks is first strained, to remove sand and sediment, and the resultant oily mixture is then heated to about 180° F. The remaining dirt and sediment is then allowed to settle out, and the oil which has been floated off is centrifuged for final purification. G. S.

See also Abstracts Nos. 129, 143, and 147.

Lubricating Oil and Wax.

143. Process Conversion of Cylinder Stock Sludge to Plant Fuel Oil. L. W. Barbour. American Petroleum Institute Bulletin, 1929, **10**, No. 2, 15–17.

Acid sludge from the treatment of cylinder oil stock at 160° F. is pumped through strainers into an equal quantity of fuel oil, which has previously been brought to 120° F. with simultaneous agitation. Air agitation is continued during the addition of the sludge and for a further 5–10 mins. A pump is now employed to circulate the fuel oil-sludge mixture, drawing from the bottom of the kettle and discharging into the top, the temperature

being meanwhile raised to 180–200° F. by means of closed steam coils. Occasionally the sludge cannot be broken by means of air, which is then replaced by steam. A hard sludge which tends to coke readily and is difficult to disintegrate is formed if the acid treatment of the cylinder oil stock is carried out above 160° F. Moreover, sludge produced at the correct temperature will coke if run into fuel oil which is too hot. More fuel oil (125 bbl.) is required to disintegrate the sludge (40 bbl.) from the second acid treatment of the cylinder stock. During storage the sludge fuel is kept agitated by means of a small quantity of live steam and by pump circulation; it is gravity-fed to injector type oil burners. W. S. N.

144. Eight Experiments on Commercial Lubricants at the United German Foundry, Düsseldorf. G. Baum. *Petr. Zeit.*, 1928, **12**, 514.

The above-mentioned company has undertaken research on the durability of normal turbine oils. Eight turbines, the smallest of 2300 KW power, and the largest of 5700 KW power, were submitted to the test.

One German, one S. American, three Russian and one Pennsylvanian turbine oils were selected whose viscosities lay between 3·04 and 3·56 E at 50° C. The following conclusions were drawn from the results obtained:—

1. Humidity could not be determined in measurable quantities in the circulating oil.

2. Oil sludge was observed in one case and with the S. American oil after 2,000 working hours.

3. Hard asphalt constituents, soluble in oil but insoluble in benzine, were not observed in any case.

4. Emulsion formation was absent in fresh oil and in used oil. Even the oil separated from the oil-sludge was not emulsified in the experiment.

5. Variation of specific gravity and flash point during the period of use was too small to give any conclusion as to the quality of the oil.

6. A uniform increase in the viscosity of the experimental oil was observed in all cases.

7. The ash content of all fresh oils was under 0·05 per cent. and increased gradually. In one case, the ash remaining from a larger sample was analysed, and gave traces of SiO_2, Fe_2O_3 and Al_2O_3 20 per cent. (chiefly Fe), CuO 63 per cent.; traces of CaO, MgO and SO_3, ZnO and PbO were absent.

8. The acid value lay, with fresh oils, between 0·028 and 0·11. The increase was not uniform and appeared to signify ageing in practice.

9. A large increase of saponifiable constituents occurred in some oils. The value of the acid constituents obtained by means of the saponification number and acid value appeared to be a marked sign of ageing of the oils.

10. Tar number was very favourable in the case of fresh oils. The behaviour in practice showed that the tar number could not be of great importance in the judgment of oils in the case of fresh oils.

The German and S. American experimental oils were not verified, so that two experiments had to be abandoned after 2000 and 3700 working hours, but all Russian and Pennsylvanian oils were well proved. The experiments still remain unfinished although one experiment lasted 20,000 working hours. W. S. C.

145. A Study of Water-Miscible Mineral Oil Preparations. R. Hart. *Ind. and Eng. Chem.*, 1929, **21**, 85.

The water-miscible or "soluble" mineral oils used in textile manufactures and as cooling and lubricating agents in metal work are clear, oily liquids that give spontaneous stable emulsions with water. The principal ingredient of the soluble oils is a mineral oil, usually a low viscosity spindle oil. The

emulsifier may be (*a*) saponified resin, oleic acid or other fatty acids with some alcohol; (*b*) sulphonated animal or vegetable oils with or without alcohol. A sulphonated mineral oil obtained as a by-product from the manufacture of medicinal paraffin can also be employed.

Two types of soluble oils were investigated. In one case the emulsifier was the sodium soap of oleic acid and in the other sulphonated castor oil (Turkey red oil). The mineral oil used had S.G. 0·847 and a viscosity of 82 seconds Saybolt at 100° F.

The relation between the proportions of oleic acid soap, free oleic acid and the mineral oil were studied. As the hydrated soap is sparingly soluble in the mineral oil, the addition of a small amount of alcohol is necessary to produce a uniform product. The soap emulsifier consisted of oleic acid 30 grams, 30° Bé, caustic soda solution 15 c.c. and alcohol 30 c.c.

The quantities of mineral oil which can be added to this amount of emulsifier are very small and a homogeneous product is not produced unless free oleic acid is added as well. For example, the addition of about 25 grams of oleic acid will permit about 800 grams of mineral oil to be added to the mixture containing the above amount of emulsifier to produce a clear soluble product. A miscibility diagram for the soap, mineral oil, oleic acid mixtures is given in the original.

The relation between the quantities of sulphonated castor oil, mineral oil and the necessary amount of oleic acid, firstly, to give clear products, and, secondly, to produce a cloud were also studied. The results are tabulated and also depicted on a triangular diagram.

Increasing the proportion of oleic acid in a soluble oil caused a decrease in the permanency of the emulsion which it gives with water. It was found that as long as less than 68 grams of free oleic acid was present per 100 grams of saponified oleic acid, the emulsions were satisfactory. When working with soluble oils it is important to establish certain criteria—viz.: (*a*) how much oleic acid is required to produce a clear mixture; (*b*) how much oleic acid the mixture will tolerate and still give a good emulsion; (*c*) the difference between the first two items; (*d*) how much mineral oil can be added and still obtain a good emulsion. Methods of determining these quantities, and also the effect of additions of alcohol and alkali, are discussed, and suitable tests for determining the stability of the products are described. R. W. L. C.

146. Cutting and Quenching Oils. C. H. Hudson. *Trans. Junior Inst. Eng.* **39**, 215.

The main duties of a cutting oil are those of lubrication, the dissipation of heat and the cleaning of the work. They should have no tendency to cause rusting or to turn rancid and should be antiseptic.

Cutting oils may be divided into two classes: soluble cutting oils and straight cutting oils.

Soluble cutting oils generally consist of a vegetable or animal oil, saponified with caustic soda or potash, with which mineral oil has been blended to produce a homogeneous liquid or paste. The product should give a permanent emulsion with many times its weight of water.

Straight cutting oils consist of lard oil, sperm oil, mixtures of lard oil and mineral oil, cotton seed and mineral oil, deflocculated graphite and mineral oil. The free fatty acid content should not exceed 5 per cent.

The methods of application of cutting oils and the suitability of different oils for various purposes is dealt with. The advantage of using straight cutting oils instead of soluble oils in machine shop practice is pointed out. The former class of oils enable work to be carried out more rapidly, as they

permit the feed and spindle speed to be increased by 20 per cent. without detrimental effect on the life of the tool.

The desirable characteristics of a good quenching oil are low volatility, high specific heat, as slight a tendency to oxidation as possible and maximum conductivity for heat. The character of the quenching oil depends to some extent on the class of work which is being hardened. The most satisfactory results for the rapid extraction of heat from the work have been obtained by using a Pennsylvanian light mineral oil blended with $7\frac{1}{2}$ to 10 per cent. of whale oil or wool oleine. The rates of cooling of a "piece" in straight mineral oil and in a blended oil show the advantage of the latter.

R. W. L. C.

147. Utilisation of Heavy Oil Acid Sludge. H. G. Osborn and L. L. Davis. American Petroleum Institute Bulletin, 1929, **2**, 4–14

"Green sludge" from the acid-treatment of cylinder stock is agitated by means of open steam in the presence of: (1) acid oil or "top" oil recovered during a previous run; (2) acid sludge from the refining of Dubbs pressure distillate; (3) water, and (4) solvent oil (gas oil). Three layers are obtained: (1) dilute acid, which is concentrated in vacuo to 63–64·5° Bé; (2) a middle layer of wet sludge or acid "coke," which is plastic at 200° F. and sets at 150° F. to a solid which does not soften on re-heating; (3) an upper layer of "top" oil, part of which is kept for use in treating the next batch, the remainder being burnt in an ordinary fuel system. After being freed from acid as completely as possible, the wet sludge or acid coke is burnt in a Duncan rotary burner, the plastic mass remaining after the elimination of the more volatile constituents being agitated in the burner by jets of high-pressure air or steam. Sufficient primary air (supplied by a forced-draught fan and, below the fuel level, by tuyeres) may be used for practically complete combustion, or the burner may be operated as a gas producer, secondary air being then used in the furnace proper.

W. S. N.

148. Contributions to the Measurement of the Viscosity of Lubricating Oils. Part I. W. Ostwald and A. Föhre. *Colloid Zeit.*, 1928, **2**.

Three kinds of flow of liquids through tubes are now recognised: (1) the laminar form of flow, which obtains during the slow flow through capillaries, for which the Hagen-Poiseuille's equation is applicable; (2) the turbulent flow for which the equation of Osborne Reynolds-Blasius serves; (3) a structural flow which is characteristic of colloidal systems under small pressure differences to which a law very similar to that characterising turbulent flow is applicable. For these cases we have the following conditions: where η rel. represents the relative viscosity co-efficient, t the time of flow, and p the driving force:—

(*a*) Small pressures (and velocities): Structural

$$p^n t = k_1\,;\ \eta\,rel.p^{n-1} = k_2\,;\ n > 1$$

(*b*) Moderate pressures: Laminar

$$p \cdot t = k_3 = \eta\ rel.$$

(*c*) Great pressures: Turbulent

$$p^{\frac{1}{n}} t = k_4\,;\ \eta\,rel. = k_5 p^{\frac{1}{n}}\,;\ n > 1.$$

These relations are shown graphically on a single diagram.

The curve connecting relative viscosity (as ordinates) and pressure or driving force (as abscissa) is in three branches with two alterations in

direction. Firstly, a steeply downward sloping line in the region of low pressures and structural viscosity; secondly, a centre horizontal portion in the region of moderate pressures and laminar flow in which the Hagen-Poiseuille relation applies and a steeply upward sloping portion in the region of turbulent flow. W. Ostwald and his co-workers have been able to realise this general viscosity curve in one and the same solution—*e.g.*, in the cases of gelatine, ammonium oleate, cotton yellow, etc.

It appears that in all the hydrodynamic theories of lubrication, the condition of laminar flow is made the basis of argument and in all accurate conventional measurements of viscosity are made under laminar flow conditions. However, the field of turbulent flow is of special interest for all lubricating oils which are employed for the lubrication of rapidly moving machine parts, such as the pistons of internal combustion engines at high speeds. The authors are not aware that the presence of turbulence has been established or that the figures for the Reynold's criteria for different oils are available. The field of structural flow is important as some lubricants are colloidal in nature. Up to the present the general validity of the laminar form of flow in the theory of lubrication appears rather to be assumed than experimentally verified.

The experimental part of the paper deals with the determination of the viscosity of 17 different oils at 25° C. under pressure heads of 5·5 to 101 cms. of water. Eleven observations were made at different pressures with each oil. The essential parts of the apparatus employed consisted of a capillary viscometer immersed in a thermostat bath connected by an air tube with a wide bore U-tube used as a source of measurable pressure having one limb of one metre and the other limb two metres in length. The oils examined comprised light and heavy machine and steam cylinder oils as well as three samples of Voltols. Full results are tabulated, viz.: the pressure p, which is the sum of the manometer pressure plus the mean hydrostatic pressure of the liquid in the viscometer, the time of flow t, and the product pt, which, according to Poiseuille's law, should be constant. The method was verified by carrying out a series of observations on glycerine with the result that the maximum variation of the product pt was only 0·68 per cent. from the mean value. The general results show that with few exceptions all the 17 oils examined behave like glycerine under the experimental conditions, and that at 25° C. they follow the Hagen-Poiseuille law, giving substantially constant values for the product pt. This was not expected, as it was thought that, among the lubricating oils examined, a system closely related to the colloids would be found. In the case of the second Voltol, slight evidence and in the case of the third and most viscous Voltol (Voltolgleitol VI.), very distinct evidence of structural flow was observed—*e.g.*, the product pt, at low pressures was greater than at higher pressures. The results for this Voltol are compared with those of a 6·53 per cent. solution of caoutchouc in toluol. The product pt, for the caouchouc solution falls from 5592 at 10·77 cms. pressure to 3053 at 101·77 cms., while that for the voltol decreases from 3423 to 3157 under the same conditions.

The influence of temperature on viscosity was also investigated in a similar apparatus to that used for the pressure viscometry. All the observations at different temperatures (20°–100° C.) being made under a pressure head of 7·5 cms. of water. The results show that Voltols have a somewhat smaller temperature co-efficient than other lubricating oils.

Good agreement between the observed and calculated results for the effect of temperature on viscosity was obtained by use of a modified form of Schwedheim's equation. R. W. L. C.

149. CONTRIBUTIONS TO THE MEASUREMENT OF THE VISCOSITY OF LUBRICATING OILS. Part II. Wo. Ostwald and A. Fohre. *Colloid Zeit.*, 1928, **3**.

It is a well-known fact that mechanical treatment alters the viscosity of many colloidal solutions. Hence the effect of mechanical rubbing or grinding on the viscosity of lubricating oils has now been studied. The method employed consisted in rubbing the oil in a mortar with a mechanically operated pestle for a number of hours. The object being to determine whether or not the viscosity of an oil is likely to undergo alteration during the actual rubbing to which it is subjected in a bearing. The viscosity of the oil was determined, before and after the mechanical treatment, in a capillary viscometer under various pressure heads by the method described in the preceding abstract. Four oils were tested after 24 hours rubbing. In the case of three of them—viz., a heavy machine oil, an automobile oil and a machine oil, the agitated oil had a higher viscosity than the untreated oil. The product *pt.* (pressure × time of flow) showed an increase of about 3 per cent. in the case of the machine oils and 29 per cent. in the case of the automobile oil. The influence of stirring for periods of from 20 to 120 hours on the viscosity of a saturated steam cylinder oil was also investigated. The viscosity of the oil was reduced by stirring for 20 hours and then rose to a maximum after 100 hours, which was slightly less than the original viscosity. After stirring for a further period of 20 hours, making 120 hours in all, the viscosity fell off again. The possibility of the increase in viscosity on rubbing being due to chemical change and polymerisation as well as a mere mechanical effect is discussed.

The effect of the addition of various substances on the pressure viscosity of lubricating oils was investigated with the following results: the addition of acetic acid and oleic acid (2·7 per cent. in each case) was found to cause a marked reduction in the viscosity, acetic acid having the greater effect. The addition of alcoholic caustic potash solution in general caused a rise in viscosity, while two samples of "Voltol" to which this reagent was added exhibited structural viscosity to a marked degree. The addition of small amounts of highly dispersed graphite and talc, and also soap and lanoline was found to raise the viscosity of mineral oils. The "Voltols," on the other hand, show a reduction on the addition of soap. R. W. L. C.

150. THERMAL PROCESSES IN PETROLEUM REFINING. The Edeleanu Process and its Technical Development. R. Plank. 24th Ann. Meeting Am. Soc. Refrig. Engineers, N.Y. Aug. 5, 1928. (*C.f.* Abs. 637, Vol. 14, 320A.)

When liquid sulphur dioxide is added to a mineral oil distillate the SO_2 is dissolved until the state of saturation is reached. The amount necessary for saturation increases with the temperature, and when saturation is reached further addition of SO_2 produces the formation of a second layer. The fact of great importance discovered by L. Edeleanu is that the selectivity of the dissolving power of SO_2 increases rapidly as the temperature falls. Aromatic and unsaturated hydrocarbons are soluble in every proportion at high and low temperatures alike. The saturated hydrocarbons are soluble with difficulty at room temperature and hardly soluble at all at 14° F. The solubility of the naphthene hydrocarbons is small at 14° F., but increases with temperature more rapidly than in the case of the saturated hydrocarbons. Therefore it may be concluded that the separating process will be most satisfactory when carried out at a low temperature and apart from the higher yield of refined oil, the employment of low temperatures has the

added advantage that less SO_2 is needed. According to the composition of the distillate from 50 to 100 per cent. more SO_2 is required at 50° F. than at 14° F.

Distillates rich in aromatic hydrocarbons show a marked improvement after treatment with SO_2, and in the case of certain kerosines the illuminating power can be almost doubled. The use of the sulphur dioxide refining process is not limited to kerosine distillates. In every case a separation of the hydrocarbon groups is brought about. Thus from the benzine fraction an extract (portion soluble in SO_2) is obtained from which quantities of benzine and toluene can be recovered by distillation.

The sulphur dioxide refining process also effects the removal of a considerable amount of the sulphur compounds, amounting in some cases to as much as 90 per cent. of that in the original distillate.

The following table gives the effects of refining distillates with SO_2 at 50° and 14° F. respectively on the specific gravity of the products and the volumes of refined oil and extract.

SO_2 Treatment of Distillates.

Yield = per cent. on distillate treated.

Origin.	Crude oil.	Distillate.	Treatment at 50° F.				Treatment at 14° F.			
			Refined Oil.		Extract.		Refined Oil.		Extract.	
	S.G.	S.G.	Yield.	S.G.	Yield.	S.G.	Yield.	S.G.	Yield.	S.G.
Bustenari	·859	·821	66·6	·808	33·4	·848	75·0	·805	25·0	·866
Tintea	·899	·822	74·6	·812	25·4	·850	82·5	·810	17·5	·874
Mexico	·883	·804	82·8	·795	17·2	·864	84·0	·794	16·0	·865
Peru ..	·858	·819	79·9	·813	20·1	·853	89·5	·814	10·5	·864
Balachany	·874	·820	87·4	·814	12·6	·853	93·1	·816	6·9	·893

Sulphur dioxide extracts resinous and asphaltic products as well as aromatic hydrocarbons from lubricating oil distillates.

The technical process is carried out on a commercial scale in the following manner. As sulphur dioxide rapidly attacks metals in presence of water, all moisture must be carefully excluded. Therefore, the distillates are dried in salt filters and drying towers. The mixing process is counter-current. The oil cooled to 14° F. enters the mixers at the bottom, and the heavy SO_2 also cooled to 14° F. near the top. In the course of its progress upwards the distillate meets fresh quantities of SO_2 descending. Finally the light refined oil forms a layer in the upper portion of the mixer, while the heavier extracts are at the bottom. In the case of light oils this mutual penetration suffices, but for operating with heavy oils the mixers contain ring packing or are provided with a stirring apparatus. The lighter refined oil is drawn off from the top of the mixer and the heavy extract from the bottom. Both of them, which are at approximately the same temperature at which they entered the mixer, pass to the evaporating system where the SO_2 is driven off and almost completely recovered by condensation and compression.

The thermal history of the process is as follows: The crude distillate enters the plant at about 77° F., and it is first cooled to about 32° F. by heat interchange with the mixture of cold refined oil and SO_2 flowing out of the mixer. The refrigerating machine, which employs SO_2 as the cooling medium, brings the oil down to 14° F. in the distillate cooler. The liquid sulphur dioxide is first cooled to about 27° F. by the mixture of cold SO_2 and extract leaving the mixer and subsequently to 14° F. in the SO_2 cooler by the refrigerating machine. Thus the distillate and the SO_2 enter the mixer at 14° F. The refined oil (in a particular case 77 per cent. of the

distillate), together with some 15 per cent. of SO_2, is heated on leaving the mixer to about 59° F. in thermal contact with the unrefined distillate. It then enters the refined oil evaporator, and the dissolved sulphur dioxide is driven out of the solution by heating with exhaust steam. The pure refined oil flows off, and as it is at a fairly high temperature, it is used in modern plants for heating the refined oil solution before the latter enters the evaporator.

The extract solution, which consists mainly of SO_2, is heated after leaving the mixer to about 54° F. in thermal contact with fresh liquid SO_2, and the large quantities of SO_2 which it contains are driven off as gas in the extract evaporators by heating with exhaust steam to about 140° F. The pressure of the SO_2 vapour rises at this point to about 160 lbs. The extract is then led in large modern plants through a series of evaporators, each operated at a lower pressure than the preceding one. In the fifth and sixth low pressure evaporators the SO_2 gas is drawn off by the two lower stages of the exhausting pump. The SO_2 is liquefied and returned to the cycle of operations. When issuing from the system, the finished extract, which is pumped out of the last (sixth) evaporator, contains only about ·04 per cent. of SO_2.

In the case of large plants, in which steam engines are employed to drive the compressors, the exhaust steam being used for heating purposes, 60 lbs. of steam and 0·4 kilo watt hours suffice to treat one barrel of distillate. The total output of plants operating on the Edeleanu system is about 60,000 barrels of oil per day.

The article is illustrated by photographs and diagrams. R. W. L. C.

151. Modern Problems of Engine Lubrication. L. H. Pomeroy. *Auto. Eng.*, 1929, **19**, 70.

It is pointed out that most of the scientific work on lubrication has been carried out at ordinary temperatures, with the result that much of the available information on this subject is restricted of the middle of the temperature scale. The lubricating oil, which is responsible for most of the troubles which are experienced with internal-combustion engines, may vary in the main property of viscosity by as much as 20 : 1 over a temperature range of 140° F.

The importance of supplying oil as soon as possible to the cylinder walls of an engine when making a cold start is stressed, as conditions may exist which cause extremely rapid rusting. It is even possible for piston seizure to result after a number of cold starts and very brief runs in severe weather.

In order to determine the effect of the temperature of the lubricating oil and of the water jacket on the mechanical efficiency of an engine, a series of measurements of the friction mean effective pressure were carried out on a Sprague dynamometer over a speed range of 800–3,000 r.p.m. These measurements were made as nearly as possible in two ways, with varying oil temperatures and constant cooling water temperatures and vice-versa. The tabulated results and curves given in the original show the dependence of the friction on the oil and water temperatures. The fall in the friction follows to some extent the fall in the viscosity of the lubricant, but the latter is vastly the greater over a given temperature range.

The fuel consumption of the engine at part throttle, 1000 r.p.m., and at varying oil and water temperatures, was also determined. The engine was started cold, and then allowed to run with the same throttle opening until the oil and water had become thoroughly hot. The brake mean effective pressure rose from 10·7 lb. per sq. in. at a mean oil and water temperature of 100° F. to 15·6 lb. per sq. in. at 152° F., while the fuel consumption dropped from 1·95 to 1·28 lb. per brake horse power per hour. It is suggested that

the thermal efficiency is affected but little by the water jacket temperatures, and that the saving on account of thermostats and radiator shutters is caused almost entirely by the reduction in the oil viscosity.

The questions of dilution, carbonisation, corrosion due to the deposition of water in the sump, and the efficacy of air filters, are dealt with at some length and are the subjects of some practical observations. R. W. L. C.

152. STUDIES ON CRYSTALLISATION OF PARAFFIN. Y. Tanaka, R. Kobayashi and S. Ohno. *J. Fac. Eng. Tokyo* 1928, XVII., **15**, 275.

Part I.—The Crystal Forms of Paraffin from Shale Oil.

The crude paraffin used was obtained by cooling and pressing a distillate, boiling at over 300° C. at ordinary pressures, from crude Fushun shale oil. The crude paraffin, which had M.P. 56° C., S.G. (d73/25) 0·7875 and refractive index (n 73/D) 1·4362, and contained about 12 per cent. of oil, was crystallised from a mixture of alcohol and ether. The product was fractionally sweated, the sweating temperature being raised 0·5 or 1·0° C. in steps to produce the successive fractions. The melting points of the fractions rose from 56·3° to 64·0° C.; the specific gravities from 0·7751 to 0·7810, and the refractive indices from 1·4285 to 1·4311 in regular increments. This fact and the similarity of these values to those of the normal paraffins observed by Krafft, show that the major portion of the shale paraffin consists of normal hydrocarbons. In general, the paraffin crystallises from its more concentrated solutions in alcohol in needle forms and from weaker solutions in plate form. Slow cooling favours the formation of plate crystals.

The plate formation of paraffin crystals is considered to be the most stable arrangement of the paraffin molecules. The difference in crystal type depends upon the cooling velocity and the viscosity of the medium and not upon the chemical nature of the paraffin. This is proved by the fact that needle crystals can be obtained from the plate crystals of shale paraffin by recrystallisation from a highly viscous mineral oil, and plate crystals are obtainable from the solution of the needle crystals of petroleum paraffin dissolved in a fluid oil. The simplest form of paraffin crystal is a perfect rhombic plate, having angles 110° ($\pm$1°) and 70° ($\pm$1°), which was observed at the highest dilution and the lowest cooling velocity of the solution. This form becomes more complicated with increasing concentration and cooling velocity. The complicated plate forms are considered to be produced by parallel growths of the rhomb in the direction of the 110° angle. Examination of the growth of needle crystals under the microscope showed that they were produced by successive parallel growths of the rhombic plate in the direction of the 70° angle. Diagrams and photomicrographs of the paraffin crystals are given.

Part II.—Crystal System of Paraffin from Shale Oil.

The crystal form of shale paraffin has been further studied. The fundamental form is a rhombic plate with angles of 110° and 70°. It exhibits double refraction and parallel extinction. Examination in polarised light shows that the three crystal axes are parallel to the three vibration axes. Complicated plate forms of shale paraffin also conform to the characteristics of the rhombic system crystallographically and optically. Needle crystals show the same optical properties as the plates, but differ from the plate form in the angle of the optic axes.

Part III.—Crystal Forms and System of Petroleum Paraffin.

The paraffins used for this investigation were derived from petroleums of Echigo, Formosa and California. The paraffins of Echigo and California crystallise in needle form from the paraffin distillates and those of Formosa in laminar form. The latter becomes similar to the others after being melted and solidified. The physical properties of these paraffins do not show any

marked differences (S.G. at 73/25 0·7781–0·7841; Refractive index 1·4288–1·4299; M.P. of fractions 53·6°–56·5° C.).

A low viscosity of the medium favours the formation of plate crystals and a high viscosity needle crystals. The pressed distillate from the Formosa oil, which deposited paraffin in plate form, was of lower viscosity than those from the other two crudes referred to. The crystal system of various forms of the petroleum paraffins was investigated, and the results show that the crystals belong to the rhombic system. The simplest form obtained from alcoholic solution was a perfect rhombic plate having the same angles and characteristics as that obtained from shale paraffin, and more complicated forms may be considered to be produced by parallel growths of the rhombs as was seen in the case of the shale paraffins. Both petroleum paraffins of plate and needle form are transmutable into each other by changing the viscosity of the petroleum distillates (crystallising media). Photomicrographs of petroleum paraffins crystallised from alcoholic solution are given.

R. W. L. C.

153. Data on the Solubility of Paraffin Wax. Weber and Durslap. *Oil and Gas J.*, 1928, 46, 154.

Previous work on this subject has shown that: (1) The solubility of paraffin waxes increases as the melting point of the wax decreases. (2) The solubility decreases with increasing viscosity of solvent. (3) Variations in solubility due to differences in melting point decrease with increasing viscosity of the solvent, and therefore with decreasing temperature. For the present work, recrystallised paraffin was wax used, having the following constants: melting point 56° C., density ·775 at 20/4° C. The hydrocarbons used as solvents were:—

	B.P.	Sp. Gr.
n-pentane	36·1–36·3	·631
n-hexane	68·9–69·2	·661
n-heptane	98·2–98·4	·684
n-octane	124·5–124·6	·706
iso-decane	159·8–160·1	·721

The following summarises the results obtained:—

Temp. ° C.	Gms. paraffin/100 c.cs. of solvent.				
	Pentane.	Hexane.	Heptane.	Octane.	Iso-decane.
0	—	2·77	1·37	·99	—
5	—	3·69	2·18	1·69	·94
10	5·11	4·81	3·55	2·90	1·44
15	6·94	6·07	5·06	4·24	2·74
20	9·53	8·31	7·18	5·93	4·98
25	17·16	16·23	14·36	11·66	9·17

A. R. S.

154. Separating Paraffineous Constituents and the Like from Oils. Aktiebolaget Separator-Nobel. E.P. 276,658. Aug. 13, 1927.

In order to separate components normally heavier than the oil, there is added to the oil, matter such as trichlorethylene, which will make the specific gravity of the oil-mixture greater than that of the heavier component. The mixture is then separated in a centrifuge which is provided with a mechanical device whereby the separated component is conveyed to the outlet G. S.

155. Treatment of Hydrocarbons with Liquid Sulphur Dioxide. *Allgem. Ges. f. Chem. Ind. m.b. H.* E.P. 281, 337. Feb. 14, 1929.

In the treatment of hydrocarbons with liquid sulphur dioxide, the latter is recovered by the evaporation in stages of its mixtures with hydrocarbon constituents, the low pressures necessary for efficient evaporation in intermediate and final stages being obtained by a jet apparatus operated by the sulphur dioxide vapour from the first-stage evaporators, which are heated to give the increase of pressure necessary to work the jet. The reduction in pressure obtained by means of the jet is also employed to remove sulphur dioxide vapour from the cooler, the liquid sulphur dioxide in which is thus brought down to the temperature required for treatment of the oil.

W. S. N.

156. Art of handling Heavy Oil Residues. O. E. Bransky and F. M. Rogers, Assrs. to Standard Oil Co., Indiana. U.S.P. 1,698,471. Jan. 8, 1929.

Residual products resulting from the non-cracking distillation of asphaltic base crude oils, and representing not more than 10 per cent. of the original crude oil, are diluted with 7–9 parts of a light hydrocarbon oil having its end b.pt. within the gasolene range and after standing, the undissolved asphaltenes are removed by decantation or centrifuging before treating the remaining diluted oil with sulphuric acid in accordance with U.S.P. 1,698,452.

H. S. G.

157. Process for Reclaiming Used Lubricating Oil. G. L. Chewy. U.S.P. 1,698,257. Jan. 8, 1929.

Used lubricating oils, containing substantial quantities of fine dirt and water, are reclaimed by treating them with dry ground caustic soda. The treatment is continued for from ten to fourteen hours at a temperature of 210° F. ± 5° F. By this process the emulsified dirt and water are slowly removed from the oil.

H. E. T.

158. Process of and Apparatus for Recovering Paraffin. Gruse and Faragher. U.S.P. 1,663,592. March 27, 1928.

Slack wax is treated in a tank containing water at regulated temperature by being forced along helical inter-meshing screw conveyors so that it is subjected to continual cutting and pressing. By this means, the softer low-melting wax and entrained oil are floated to the top of the tank by the water, while relatively high-melting oil-free wax passes out of the other end of the conveyor.

A. R. S.

159. Treating Hydrocarbon Oils. F. W. Hall. U.S.P. 1,700,479. Jan. 29, 1929.

The process, which aims at effecting economies in the use of decolorising clays, consists first of all, of using the clay to remove part of the colour from a light oil, such as a spindle oil, the oil and the clay being agitated together at 250° F. until the clay is exhausted. The mixture is passed through a filter press, and the clay cake, which may, if desired, be first washed with naphtha to remove the bulk of the oil, is then revivified in the press by treatment with superheated steam. The revivified clay is then found to have only recovered a portion of its efficiency towards light oils, but nevertheless now possesses most of its original efficiency for the decolourisation of heavier and darker oils. The revivified clay is then used to decolourise a heavy lubricating oil, agitation being carried out with the oil at 300° F. The spent

clay from this second operation is again revivified and used for treating very dark coloured viscous residue oils such as cylinder stock, the treatment being carried out by agitation at 450° to 500° F. After the clay has been exhausted on the darkest coloured oil, it may be fully revivified to an efficiency approaching its original activity by treatment with solvents or burning and used again in a fresh cycle of operations. R. W. L. C.

160. Bleaching Montan Wax. W. Pungs and T. Hellthaler, Assees. to I. G. Farbenind. A.-G. U.S.P. 1,698,450. Jan. 8, 1929.

Crude or deresinified montan wax is bleached by exposing it, either in solution in a volatile organic solvent or as a finely divided suspension, to the anodic action of an electric current of 300–500 amps. per sq. metre of anode and 3–5 volts., in the presence of electrolytes such as 40 per cent. sulphuric acid, and oxygen transferrers—*e.g.*, compounds of chromium, cerium, vanadium, manganese and the like, while stirring and heating the mixture to about 100° C. Lead is a suitable electrode material, and the anode is preferably separated from the cathode by a porous diaphragm or solid impermeable material such as glass which extends not quite to the bottom of the electrolyzing vessel. H. S. G.

161. Decolorizing or Bleaching Montan Wax. J. Y. Johnson. From the I. G. Farbenindustrie A.-G. E.P. 302,626.

A process is claimed for decolorizing or bleaching montan wax by means of chromic acid in sulphuric acid solution in the absence of acetic acid or other organic solvents. It consists of suspending crude or deresinified montan wax in a solution of sulphuric acid and/or a warm solution of a chromic salt, for which purpose a spent solution of a previous bleaching operation is preferably employed, and adding a solution of chromic acid. The mixture is heated to 100° C. or above, simultaneously evaporating water and periodically drawing off the spent solution during the bleaching operation. The bleached wax is allowed to remain in a warm state until complete separation has taken place. H. S. G.

162. Valuable Products from Montan Wax. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 305,552. Feb. 5, 1929.

The process of bleaching crude or deresinified montan wax by means of oxidising agents, in particular chromic acid, is already known (*cf.*, E.P. 289,621, 299,133). A particularly valuable product, yellow to white in colour and of high saponification value, is obtained by treating montan wax (1 part) with chromic acid, in the presence of at least 5 parts of a mineral acid (sulphuric or phosphoric) in the form of a 30–70 per cent. aqueous solution, at 100–130°. Simultaneously with the bleaching action, saponification of the wax esters is effected. The amount of chromic acid used and the duration of the process are chosen to substantially avoid carbonisation. W. S. N.

163. Manufacture of Lubricating and Insulating Oils. J. Y. Johnson, Assr. to I. G. Farbenind. A.-G. E.P. 305,553. Feb. 5, 1929.

Lubricating and insulating oils are prepared from substances such as coal, mineral oil, tar, etc. If the starting material is solid it is destructively hydrogenated or extracted by means of a solvent; if liquid, it is destructively hydrogenated or cracked. The liquid products, particularly those of the middle-oil type, are exposed in the coherent liquid phase to the action of high-tension, preferably high-frequency electric currents. *E.g.*, brown coal is hydrogenated at 450° and 200 atm., and, of the liquid product, a fraction

of b.p. substantially up to 300° (viscosity 2·7° Engler at 50°) is subjected to a treatment with a current of about 10,000 volts and 500 periods. After separating the viscous products by distilling off the more volatile constituents, the latter may be treated again. W. S. N.

164. Art of Distillation. W. K. Lewis and N. E. Loomis, Assrs. to Standard Oil Development Co. U.S.P. 1,697,195. Jan. 1, 1929.

High boiling-point oils are distilled in one or more units of a battery by direct fire heat under a low absolute vapour pressure and below a temperature at which substantial cracking occurs. The vapours are removed through a passage without constriction between the surface of the oil and a condensing column from which the condensate is collected. Drop in the temperature of the vapours until reaching the condensing zone is prevented by jacketing the passage with a hot medium, thereby preventing refluxing. H. S. G.

165. Manufacture of Lubricating Oils. L. Mellersh-Jackson, Assr. to Sun Oil Co. E.P. 305,846. Feb. 14, 1929.

Lubricating oil is produced from crude mineral oil by distilling off the lighter fractions under limited temperature and pressure in the absence of oxygen, treating the lubricating stock with aqueous soda, removing the water and certain oil fractions under reduced pressure, and bringing the residue distributed over an extended surface into heat exchange with mercury vapour, the temperature of the latter and the pressure on the oil being limited to minimise cracking, the condensed lubricating oil distillate being then freed from malodorous volatile constituents by being distributed over an extended surface whilst subjected to a high vacuum. W. S. N.

166. Apparatus for Reclaiming Used Lubricating Oil. P. F. Miller. U.S.P. 1,694,478. Dec. 11, 1928.

This invention relates to an improved and simplified apparatus for the purification of used lubricating oils. The treatment of the used oil, in order to remove solid impurities, water and diluents, consists of three main operations.

The used oil is first circulated through a heater and is then passed through a centrifuge in order to remove water and any gritty particles which may be present. A counter-colloid such as trisodium phosphate, oleic acid or soda-ash is now added to the oil, causing the finely divided carbon suspended in the oil to become agglommerated. The oil is again centrifuged when the carbon is completely removed. The oil is finally circulated through a tower having a series of baffle plates and charcoal beds, which break the stream of of oil up into thin layers. Warm air is then continuously blown through the oil, until all the undesirable diluents have been removed.

It is claimed that by this process a recovered oil may be obtained which is as good, if not better than the original unused lubricating oil. H. E. T.

167. Apparatus for and Method of Treating Oil. E. S. Pearce. U.S.P. 1,698,273. Jan. 3, 1929.

A method is described for treating oils adapted for lubricating the journals of car axles, whereby the oil is thoroughly cleansed of foreign matter, such as carbon, dirt, water and heavier greases.

The apparatus consists essentially of a treating tank, provided with a series of slanting baffle plates. Between one edge of each baffle plate and the adjacent edge of another there is a passageway. The upper end of the tower terminates in a tapered cone, having an opening from which a discharge pipe leads. The purified oil is drawn off from the treating tank at a point

intermediate between its ends, and passes to a clean oil tank. Each baffle plate is provided on its underside with crosswise baffle bars, serving to interrupt the flow of materials such as dirt and water, having a specific gravity greater than that of the oil. During the upward flow of the oil which is given a preliminary heat treatment in order to reduce its viscosity, the impurities are deposited on the underside of the baffle plates. The water in the oil passes out of the conical top of the tower as steam, while the purified oil is drawn off from the tower at a point below the steam outlet, but above the baffle plates. H. E. T.

168. Bleaching Montan Wax. W. Pungs and I. G. Farbenind. A.-G. U.S.P. 1,699,250. Jan. 15, 1929.

Crude or deresinified montan wax is bleached by treating with an aqueous solution of chromic acid at 102° C. in the presence of solutions of organic acids such as acetic or benzine sulphonic acid. H. S. G.

169. Treating Hydrocarbon Oil Residues. F. M. Rogers, Assr. to Standard Oil Co., Indiana. U.S.P. 1,698,452. Jan. 8, 1929.

Residues from the non-cracking distillation of asphaltic base crudes are mixed with a light hydrocarbon oil such as kerosene or gasolene and treated with 66° Beaumé sulphuric acid. The admixed oil is then removed and the residue may be further treated by dissolving in a suitable solvent and filtering through Fuller's earth, or chilling and settling for the removal of petrolatum in order to produce high flash point heavy lubricants. H. S. G.

170. Improvements Relating to the Refining of Petroleum Oils. The Sharples Specialty Co. and L. D. Jones. E.P. 280,559. Jan. 10, 1929.

A method is described for removing wax from petroleum oils containing crystalline wax alone or crystalline and amorphous wax, which consists in adding amorphous wax to the oil, so that on chilling the mixture, the precipitate thereby obtained is sufficiently plastic or amorphous and of such high specific gravity that the wax is separated from the oil upon centrifuging. For example, the wax distillate fraction obtained from the steam distillation of Mid-Continent crude petroleum, lying between 80 and 150 seconds Saybolt viscosity at 100° F., may be dewaxed according to this invention, if 85 parts of such distillate are mixed with 15 parts of petrolatum, the petrolatum being obtained from the cold settling of an acid refined, 600° F. fire test Mid-Continent residuum. The viscous and therefore valuable oil occluded in the petrolatum is also thereby recovered. R. W. L. C.

171. Improvements in the Purification of the Wax Residues of Petroleum and Shale Oil Distillation and Apparatus Therefor. Steel Brothers and Co., Ltd. E.P. 303,812. Jan. 8, 1929.

This invention deals with an improvement of the sweating pan, which is effected by providing, in the space above the gauze false bottom, a multiplicity of horizontal pipes arranged in a series of closely spaced vertical coils. These support the scale, preventing it forming a compact mass, thus facilitating rapid drainage of the oil from the scale and allowing a much greater thickness of scale than the normal six inches to be charged into the pans. During the sweating process a heating medium may be circulated through the pipes to maintain the scale at any desired temperature.

The apparatus may be enclosed in a specially constructed chamber provided with heating or cooling means, which can be employed as desired to assist the process. W. L.

172. Manufacture of Lubricating Oil. G. H. Taber, Jr. U.S.P. 1,698,076. 8.1.29

An improved method is claimed for the combined distillation and refining of lubricating oils, which consists in vaporising the lubricating oil from a body of oil containing lubricating oil constituents in a shell still or a number of shell stills operated continuously in series, in such a manner that the lubricating oil vapours pass first through a fractionating tower and then to a treating tower in which they are subjected to the action of caustic soda solution. The caustic soda solution may be of density of 10° to 20° Bé. The minimum amount should be sufficient to remove undersirable constituents, and may be from ·5 to 15 per cent. of the weight of the vapours entering the treating tower. The amount of condensate formed in the treating tower is controlled by the amount of caustic solution introduced. The caustic soda solution passing from the bottom of the tower with the oil condensate is separated and recirculated. Vapours from the treating tower may be condensed or passed to a second fractionating tower. It is claimed that the process avoids the formation of the objectionable deposits which are normally produced when caustic soda is added to a body of oil undergoing distillation, and also that very thorough contact between the oil and the soda is obtained. The process can be operated at atmospheric or under reduced pressures and with steam injection to the still. R. W. L. C.

173. Removal of Petrolatum from Oils. C. Zweig, Assr. to Standard Oil Co. U.S.P. 1,694,090. 4.12.28.

The invention provides a method of removing petrolatum from lubricating stock. It consists of diluting the stock with naphtha, chilling to about 10° F., agitating the mixture by passing gas therethrough for a few hours, and then maintaining a low temperature for a prolonged period while the petrolatum settles out. S. W. C.

Special Products.

174. Bitumen and Tar Emulsions in Road-Building. Wilhelmi. *Erdöl und Teer.*, 1929, v., 87–88.

Bitumen and tar emulsions or suspensions represent the so-called disperse systems in which the bitumen or tar is the disperse phase and water is the dispersion medium. An extremely fine distribution of bitumen and tar in water is the essential thin binding agent or medium for road-making. The following requirements are necessary in order to produce a suitable emulsion:

(1) The purest bitumen or road-tar.

(2) Stability of the emulsion to prevent decomposition and consequent lump-formation in the layer.

(3) Fine distribution of bitumen in water and insensibility of the emulsion to weather conditions.

(4) No tendency to re-emulsifying of the precipitated bitumen by the influence of water.

According to (1) the binding medium utilised in the formation of emulsions should correspond to the following German standards.

I. Bitumen.

Specific gravity at 20° C. not less than 1.0. Open flash-point at least 200° C. Maximum volatility after heating for five hours at 160° C. 3 per cent. Melting point 28–35° or 40–50° C. Penetration 60–150 or 40–80.

II. Road Tars.

	Road Tar I.	*Anthracene Oil Tar.* 50/50	60/60
Maximum specific gravity at 15° C.	1·225	1·225	1·225
Maximum ammoniacal or H_2O content	1·0% by wt.	1·0% by wt.	1·0% by wt.
Maximum content of distillates below 170° C. (light oils)	1·0% by wt.	1·0% by wt.	1·0% by wt.
Distillate between (170°–270° C.) middle oil within	12–24% by wt.	1–15% by wt.	1–10% by wt.
Distillate between (270°–300° C.) heavy oil within	4–12% by wt.	4–12% by wt.	4–12% by wt.
Maximum content of phenols	5% by volume	3% by volume	3% by volume
Maximum content of naphthalene	5% by wt.	3% by wt.	3% by wt.
Free carbon	5–18% by wt.	5–18% by wt.	5–18% by wt.
Viscosity (Hutchinson) ..	3–15 seconds	1–15 seconds	20–80 seconds
Pitch content	55–65% by wt.	45–55% by wt.	55–65% by wt.

It is known that petroleum pitches and natural asphalts, on account of their low specific gravity, emulsify easily in the dispersion medium, *i.e.* water, but tar with a higher gravity of about 1·2–1·3 is difficult to emulsify.

The emulsions are differentiated according to their production—those which require emulsifiers (stabilisers) and those which do not require such substances. The former comprise various chemical preparations, whilst the latter are prepared in specially constructed high-speed colloid mills.

The emulsifiers are mostly soaps, especially the alkali soaps of oleic acid which produce very good emulsifiers even in small quantities. The sodium compounds of the soaps show the least emulsifying effect, but potassium and ammonium compounds have the greatest emulsifying capacity. Soft bitumen with a melting point less than 25° C. is easily emulsified with a solution of water-glass. Hard bitumen is difficult to emulsify with this emulsifier.

Several patents are enumerated dealing with the application of various substances as emulsifiers. The use of naphthenic acids and of soaps together with an organic or inorganic colloid, *i.e.*, starch, is recommended. The emulsifying effect of sulphite lye in the production of asphalt and tar emulsions is also utilised. Other patents describe the application of organic sulphur compounds which accelerate the velocity of dispersion and should increase the stability of the dispersion formed. The use of ammonium compounds of acid tar and the emulsifying effects of alkaline and ammonium compounds of the oxidation products of hydrocarbons (*e.g.* paraffin) are also mentioned.

W. S. C.

175. Bitumen and Tar: Emulsions in Road-Building (*concluded*). Wilhelmi. *Erdöl und Tur.* 1929. VI. 105–106.

The application of casein solutions, starchy substances and finely divided cellulose has been detailed in certain patents as emulsifiers for tar and bitumen emulsions. Bituminous emulsions are also rendered stable by adding small quantities of materials such as glycerin, dextrin and mannite. A British patent describes the use of alkaline solutions of proteins for the emulsification

of bitumen whereby a portion can be recovered with fatty acids. Triethylamine is also mentioned. Certain American patents recommend the application of soap solutions together with colloidal clay, and of glue.

The *production of emulsions* is brought about usually in a special apparatus by which the binding medium which is to be emulsified is fused with the emulsifier, the requisite quantity of water added, and the solutions stirred vigorously. In another process the stabiliser is first dissolved in the water, and is then stirred with the molten bitumen into a special type of boiler. The relative quantities of the constituents are generally chosen so that emulsion contains 50–60 per cent. bitumen or tar and 50–40 per cent. water and about 1 per cent. of the stabiliser. The finished emulsion is dark brown or greyish-black in colour according to the substance applied and the degree of fineness.

The principal commercial bitumen and tar emulsions are described. *Euphalt cold-asphalt* is a 50 per cent. bitumen emulsion. The fineness of the emulsified bitumen particles is attained by utilising a physico-chemical process. The emulsification medium is an irreversible colloid, and is therefore insoluble in water after decomposition of the emulsion on the road.

Amongst other emulsions cited the following appears to be important :—

Teeramuls contains 90–95 per cent. tar, and 10–20 per cent. asphalt may be added if desired. The quantity of emulsifier amounts to 5–10 per cent. and the maximum water content is 2 per cent. A fractional distillation of undiluted Teeramuls gives the following results :—

Specific Gravity, 1·18.

Water, 1% (from emulsifier).

Light Oil to 170° C.	0%
Middle ,, ,, 270° C.	16%
Heavy ,, ,, 300° C.	8%
Phenols	2%
Naphthalene	3%
Free Carbon	6%
Viscosity (Hutchinson)	15 seconds.

The viscosity is increased by addition of water. This emulsion must on this account be prepared at 60°–90° C., since it is only liquid when hot but becomes gelatinous in the cold. W. S. C.

176. The Influence of Powder Fillings on the Plastic and Elastic Properties of Bituminous Masses. H. Suida and A. Schmölzer. *Petr. Zeitschrift.* 1929. XXV. 251–254.

Experiments are described in which Trinidad asphalt, coal-tar pitch and petroleum are used as the binding agent and the influence of fillers observed. The specific gravity, softening point (ring and ball method), penetration test at 25° C. and ductility at 25° C. of the binder are given :—

	Trinidad Asphalt.	Petroleum Pitch.	Coal-tar Pitch.
Specific gravity ..	1·40	1·02	1·24
Softening point ..	89·2	58·2	53·6
Penetration at 25° C.	4	27	16·0
Ductility at 25° C.	—	37	Over 100

The following fillers were utilised: Powdered crystalline limestone, quartz, granite, bricks and micro-asbestos. The five fillers gave the following results on elutriation analysis:—

	0·03–0·06 mms. % by wt.	0·006–0·03 mms. %	Less than 0·006 mms. %
Crystalline limestone	52·5	36·1	11·4
Quartz	25·5	51·9	22·6
Granite	53·7	32·4	13·9
Brick	42·3	41·3	16·4

Various graphs are given illustrating the influence of different quantities and types of fillers on the properties of bituminous substances, and the following conclusions are derived:—

1. An addition of filler changes the plastic and elastic properties of a mixture with bituminous substances. There are very few exceptions to this rule.

2. The curves for quartz and granite are practically identical, whilst in some cases those for limestone, and in almost every case asbestos and brick-dust deviate from one another and from quartz-granite curves.

3 (*a*). Penetration curves. No deviations are apparent with petroleum pitch with a limestone filler. Asbestos shows the greatest deviation. The curves diverge very sharply with increased additions of filler to the bituminous substance, but they converge to a higher penetration with asbestos than with other fillers. It appears that greater additions of such fillers would have little influence on penetration values.

3 (*b*). Ductility curves. These curves vary for petroleum pitch and Trinidad asphalt, since the latter contains a certain amount of natural fillers.

The type of filler and the size of the particles influence the properties of a road surface. The softening property of limestone as a filler in coal-tar pitch is due to the presence of reactive acid constituents in this pitch which are absent in petroleum pitch and Trinidad asphalt. The peculiarities of asbestos and brick dust are based principally on their surface structure. Asbestos differs from the other fillers by its fibrous structure which is favourable to the elasticity of bitumen. W. S. C.

177. Improved Bituminous Composition for Paving. G. Cobb. E.P. 302,808. Dec. 28, 1927.

A single layer road-paving material is described which can be adapted for use without sand or filler, consisting of 3½–5 per cent. bitumen of the type known as asphalt cement and of defined characteristics and a crushed rock mineral aggregate. Approximately ¼ gall. per sq. yd. of the bitumen is applied hot as a sealing coat after laying the above composition, and the surface is covered with clean stone screenings and thoroughly rolled.

H. S. G.

178. Purifying Dry-Cleaning Solvent. A. E. Hatfield. U.S.P. 1,704,604. March 5, 1929.

Used solvents are purified by the addition of finely divided porous cellular silica, the mixture being passed through a filter and the filtered solvent returned to the washing machine. In addition to the silica, a decolorising agent or a reagent to flocculate foreign bodies may be used. G. S.

179. Rubber-Like Substance and Process for Obtaining the Same. E. W. Hultman. U.S.P. 1,704,194. March 5, 1929.

As an example of the method of producing rubber-like substances, pressure distillate from a Dubbs cracking plant was distilled up to 205° C., and the residual oil after cooling to about 90° C. was treated with cerium oxide (1 gm. per 400 c.cs.) and agitated for a few minutes, then permitted to cool to 28° C. A gaseous mixture containing 90 per cent. carbon monoxide and 10 per cent. of methane was then passed through a catalytic mass of fused magnesium sulphate containing 1½ per cent. of finely divided nickel and a trace of palladium. The gases at a temperature of about 200° C., after passing through the above activating mass, were bubbled through the oil mixture at the rate of 2 cub. ft. per 400 c.cs. of oil. After standing for 24 hours chlorine gas was passed through to saturation, and the mixture set aside for a further period of five days. At the end of this time a dark brown coagulum had formed amounting to 15 per cent. of the original oil residue. This sticky substance was found to be susceptible to vulcanisation. R. P.

180. Production of Lamp Black. J. L. Major. E.P. 303,632. Dec. 6, 1927.

Lamp black is prepared by feeding a liquid carbonaceous substance on to trays mounted within an ignition or combustion chamber. The trays are adapted to extend one in front of that immediately above it, with cavities inclined downwardly towards the front edge of the tray at which a series of notches or outlets are provided to facilitate the cascading of the liquid from one tray to the next. Air is admitted over the respective trays through a series of orifices adapted for regulation and directed transversely across the cascading liquid. H. S. G.

181. Automobile Motor Cooling Oil. R. E. Wilson, Assr. to Standard Oil Co. U.S.P. 1,700,392. Jan. 29, 1929. Appl. April 21, 1925.

The motor cooling oil described consists of a hydrocarbon oil having a flash-point not less than 190° F., a viscosity below 45 sec. Saybolt at 100° F., and I. B. P. above 400° F. G. S.

See also Abstracts Nos. 145, 146, 173, 183, and 209.

Analysis and Testing.

182. X-Ray Quantitative Analysis of Tetraethyl Lead in Gasoline. R. H. Aborn and R. H. Brown. *Ind. and Eng. Chem.* (Analytical Edition), 1929. **1**, 26.

A successful application of X-ray methods has been made for the direct estimation of lead tetramethyl in samples of commercial gasoline. With the experimental arrangement described, an accuracy of 0·1 per gallon can be obtained, this being equivalent to a sensitivity of 1 part in 14,000. Standard solutions were made up from tetraethyl fluid of such a composition that 3 c.cs. of "fluid" contained 1·65 c.cs. of Pb $(C_2H_5)_4$. Eight samples were made up containing from 0·0 to 3 c.cs. of lead tetraethyl per gallon (3·7854 litres), commercial gasoline being used as solvent. Among the conditions which exert a marked influence on the shape of the curve obtained will be the type of radiation, and the thickness of the specimen

penetrated by the beam. From the curves obtained experimentally it appears that :—

1. The method shows great promise as an analytical tool.
2. The experimental conditions as outlined give almost straight line curves.
3. An accurate control of experimental conditions is important.

It was also found that very close current control is equally important. Moreover, the analysis as outlined can be completed in about five minutes, whereas the usual chemical method requires at least 40 minutes.

H. E. T.

183. Rapid Determinations of Specific Gravity of Semi-Solid Bituminous Substances. S. E. Berkenblit. *Ind. and Eng. Chem.* (Analytica Edition). 1929. **1**, 39.

A simple method for determining the specific gravity of semi-solid bituminous substances is described. In the method the specific gravity is obtained at 77° F. (25° C.), which is the standard temperature specified in the A.S.T.M. method of testing the specific gravity of asphalts and tar pitches, serial designation D 71–27. The method gives duplicate analyses which check to within 0·002.

H. E. T.

184. Asphalt-Precipitation Benzines. H. Burstin and J. Winkler. Erdöl und Teer. 1929. 5, 26–28, 42–44, 62–64.

For the estimation of hard asphalt (asphaltenes) in petroleum, Holde (Kohlenwasserstofföle und Fette, p. 135; Chem.-Zeitg., 1914, pp. 241–265) prescribes the use for precipitation of a colourless, pure benzine having the following characteristics d_{15} 0·695–0·705, *b.p.* 65–95° (40 cm. Le-Bel-Henninger column), not more than 2 per cent. soluble after 15 mins. shaking with a mixture of 80 parts 96 per cent. sulphuric acid and 20 parts fuming acid (20% SO_3). Kahlbaum's "Normalbenzin" conforming to this specification gives 2·81, 0·03 and 0·25 per cent. asphalt, respectively, in three heavy oils, 1, 2 and 3. Benzines, also conforming to the above requirements, are prepared by repeated sulphuric acid treatment of suitable fractions of (*a*) Boryslaw petroleum; (*b*) Grabownica petroleum; (*c*) Boryslaw natural gas gasoline; and (*d*) a cracked distillate. The asphalt contents of the same three oils using these benzines are: (*a*) 2·74, 0·00, 0·10; (*b*) 2·83, 0·035, 0·24; (*c*) 2·84, 0·025, 0·26; (*d*) 3·23, 0·16, 0·31. Thus the first is not suitable as a precipitant the second and third are equivalent to Kahlbaum's "Normalbenzin," and the fourth precipitates even more hard asphalt. None of the benzines contains any olefines or aromatic hydrocarbons, and the naphthene contents are; Kahlbaum's "Normalbenzin" 12·0; (*a*) 23·0; (*b*) 11·7; (*c*) 13·0; and (*d*) 3·0 per cent., respectively. It is therefore not essential that Pennsylvanian petroleum should be used in preparing benzine for the precipitation of asphalt.

W. S. N.

185. A Micro-Fractionating Column for Analytical Purposes. Cooper and Fasce. *Ind. and Eng. Chem.* 1928. **4**, 420.

A small round bottomed pyrex flask is attached to a column 36 cms. long, having small constrictions along its length. The bottom part of the column is vacuum jacketed and heated from outside by coils of 30 gauge Chromel wire, while the top part contains a tube supplying internal water or air cooling. The vapour temperature thermometer is entirely enclosed in the vapour space and the condensate drops from a constricted tube into the centre of a 5 c.c. burette which is water jacketed and serves as a receiver. A gas outlet is provided at the top of the receiver to which suction may be

applied. Finally the whole column is packed in a box filled with kieselguhr to ensure high heat insulation. A charge of 10 c.cs. is placed in the flask, and heating is carried out by means of a very small bunsen flame. The flow of condenser water is adjusted to obtain the desired rate of reflux. Best results have been obtained with a reflux rate of 20–40, and condensate of 2–10 drops per minute, slower distillation being required for liquids whose boiling points are closer than 10° C. A. R. S.

186. Simple Pressure Regulator for Vacuum Distillations. H. L. Cox. *Ind. Eng. Chem.* (Analytical Edition). 1929. **1**, 7.

A simple pressure regulator for vacuum distillations is described. The regulator is entirely automatic, requires a minimum amount of attention, and maintains any desired pressure within 0·1 mm. of mercury. By suitably altering the construction of the apparatus and using a pressure pump, pressures above atmospheric are readily obtained. H. E. T.

187. Sources of Errors in Sulphur Estimations in Mineral Oils. K. Fischer and W. Hess. Erdol und Teer. 1929. V. 83, 84.

Waterman's previous work on this subject had shown that solutions of mercaptans in light oils gave low results with the Engler lamp due to the formation of asphaltic bodies which remained in the wick.

In the present work analyses of ethyl mercaptan, thiophenol, thiophen and solutions of these in kerosenes were carried out in the Heslinga apparatus or when possible in the Engler lamp.

Some SO_3 is formed in the combustion of sulphur-containing oils, up to definite temperature limits. The gases pass a very hot flame zone and are then quickly cooled and absorbed in hydrogen peroxide solution. The error is great when substances of greater than 0·5 per cent. sulphur content are consumed in the Engler lamp but for kerosenes containing less than 0·05 per cent. sulphur the error is negligible.

In the Heslinga apparatus, the oil is vapourised by heating in a stream of air in a quartz tube and the gases burned and absorbed by hydrogen peroxide. No SO_3 is formed above 900° since dissociation occurs above that temperature in favour of SO_2 formation. A portion of the SO_3 is condensed in a glass tube which connects the quartz tube to the condenser. The combustion gases pass into the H_2O_2 receiver, thence through two moistened glass filters to a small absorption flask. The contents of the receiver are titrated, the condensate in the glass tube filtered and the absorption flask washed out and the contents titrated. The results with the compounds mentioned are concordant with the SO_3 estimations by the Carius method. W. S. C.

188. Auto-Ignition Temperatures of Flammable Liquids. N. J. Thompson. *Ind. Eng. Chem.* 1929. **2**, 134–139.

The experiments recorded in this paper cover one phase of a thorough investigation of the causes of fires and explosions in ovens used for drying japans, paints and lacquers.

The method of carrying out the tests was practically the same in all cases, regardless of the form of apparatus used, the ignition temperatures being determined by the appearance of flame whether the combustion was of an explosive violence or not. From the results obtained it is shown that:—

1. Copper is entirely unsuitable as an igniting surface for general test work.
2. A chromium-plated apparatus has many advantages, but does not give results in agreement with those obtained when steel is the igniting surface.
3. Steel has the most universal practical application with regard to the results obtained, although an apparatus made of steel of sufficient volume to give low results must be made to accurate dimensions.

4. Duplicate results are more readily obtained when the ignition surface is of glass, than with any of the other materials tried. Little is gained by increasing the size of the ignition chamber beyond 125 c.cs., while a decrease in the size of the flask mouth lowers the temperature of auto-ignition.

5. The lowest auto-ignition temperatures were obtained by using quantities of fuel such as to give a rich mixture in the ignition chamber. Ignition rarely takes place with quantities of fuel calculated to give mixtures leaner than the theoretical combining proportions, except at temperatures much higher than the values recorded in the tables. Hence it is concluded that relatively lean mixtures of flammable vapour and air cannot be ignited by hot metals at the temperatures commonly prevailing at burner boxes or electric heating units in japan ovens. When vapours have accumulated to a rich concentration, ignition can and does occur generally with disastrous results. H. E. T.

189. Colour-Estimating Apparatus. O. Rosenheim, E. H. J. Schuster and Tintometer, Ltd. E.P. 299,194. Oct. 31, 1927.

In an apparatus of the Tintometer type, a series of colour screens are arranged so that screens of progressively varying depth of tint may be brought in succession or withdrawn from the operative position. An index may be attached to indicate the screen in use. G. S.

190. Improved Method and Means for Determining the Content of Combustible Gas in Gas Mixtures. Siemens and Halske A.-G. E.P. 282,080. Dec. 8, 1927. Ger. Dec. 10, 1926.

An apparatus is described for determining the combustible gas content of gas mixtures—*e.g.*, flue gas, by combustion on an electrically heated test wire and by measurement of its resistance. The test wire is tightly stretched in a chamber in the direction of gas flow—*i.e.*, parallel to the longitudinal axis of the measuring chamber, between two supports made of a non-oxidisable metal such as a gold alloy, one of which is in the form of a bracket. These supports project into the measuring chamber close to the point of gas admission and form a protective zone by reason of temperature difference immediately in front of the insulating mountings. H. S. G.

See also Abstracts Nos. 134, 135, 148, 149, and 204.

Refinery Plant.

191. Thermal Characteristics and Heat Balance of a Large Oil-Gas Generator. R. D. Pike and G. H. West. *Ind. Eng. Chem.* 1929. **2**, 104–109.

This article records the results of a five-day test conducted on a large gas generator of the Jones type, one of a set of five operating at the Potrero, San Francisco, plant of the Pacific Gas and Electric Company. The gas generator consists of two connected shells which are filled with checker brick, the cycle of operations lasting 20 minutes and consisting of three periods—the make, the blow and the heat. During the make, which lasts 10 minutes, oil and steam are introduced at several points in each shell passing downward in each, the gas being removed through the offtake of the secondary shell to the wash box, where the temperature is lowered to slightly above atmospheric, the lampblack and tar being removed from the

gas stream. The blow lasts five minutes, and is intended to burn out carbon deposited in the generator during the make, the oil feed and greater part of the steam being shut off during this operation. The heat lasts approximately five minutes, while oil and air are fed in at the top of the primary and burn while passing through both shells, finally leaving at the top of the secondary shell. The heat brings the brick checkerwork up to temperature again after the slight cooling of the blow.

In order to run a heat balance on the generator, a material balance was first established. The amount of air supplied to the generator was measured with a Venturi meter located in the air supply line. The readings of the water gauge attached to the meter were checked against those obtained by means of two Pitot tubes, the results of nearly a thousand readings agreeing to nearly 1·2 per cent. The amount of oil fed to the generator was measured by oil meters which showed the quantity of oil used for heating, the quantity used for making gas and the total plant consumption. The amount of steam used during blowing and heating could not be obtained by direct measurement, and hence it was determined by analysis of the water content of the waste gases. The composition of the waste gases was obtained by Orsat analysis, and the water content by aspiration of a known volume of the gas through a calcium chloride tube. The temperature of the waste gases was obtained by means of iron-constantan thermocouples inserted in the wall of the secondary shell.

The amount of steam used during the gas making period was 14,000 pounds per hour. The temperature of the manufactured gas was obtained by means of a continuous recorder, and its composition was obtained by analysis of the gas from the entire plant. The amount of manufactured gas was calculated from the amounts of oil used in the generator and in the entire plant, together with the total plant production of gas. The composition of the oil was obtained by analysis of typical Californian fuel oil used for gas generation.

The heat balance for the blowing and heating periods of the generator shows that 85,300 B.T.U. are lost as sensible heat in the waste gases for every 1,000 cub. ft. of gas manufactured. This corresponds to a loss of 5,220,000 B.T.U. per cycle of the generator, the temperature of the waste gases varying from 784° to 797° C. By assuming an efficiency of 50 per cent. for a waste heat boiler, the installation of such a unit would allow 188,000,000 B.T.U. to be recovered for every 24 hours operation on each generator.

The heat balance for the complete generator cycle shows that 18·5 per cent. of the output is lampblack, and 3·28 per cent. tar. The average yearly operation of the producer plant calls for the continuous operation of three generators and the additional operation of a fourth generator for 12 hours per day during six months of the year.

From the experimental data obtained it is shown that the economic efficiency of the gas producing process can be materially increased, if it is possible to eliminate the production of lampblack and tar in the generator and put the heat represented by their removal back into the manufactured gas.

H. E. T.

192. Distillation and Fractionation of Petroleum. H. R. Swanson. *Petr. Times.* 1929, **21**, 443–444.

In the refining of petroleum the elimination of re-running, by taking the final cuts desired straight from the primary distillation system, does much to increase yields and to reduce chemical and treating losses. Such a system comprises a main-heating unit, heat-recovery equipment and fractionating device. For the first, the tube still has now been developed to operate with

low excess air, and consequently with overall efficiency approaching that obtained in boiler practice. For fractionation, the bubble tower has become generally accepted as most economical; towers with long rectangular nozzles and caps give somewhat better distribution of liquid on the tray than those with round nozzles and caps covering individual or grouped nozzles. For latent heat interchange the major contact between liquid and vapour is in the vapour space above each tray. Particularly when deep cuts are to be made into the crude, step-up installations comprising tube still and tower for each fraction are lacking in the simplicity and flexibility characteristic of the single-flash type of plant, which provides the solution to a previously complex problem. W. S. N.

193. Improvements in Apparatus for and Process of Distilling Hydrocarbons. C. Arnold. (Communicated from Standard Development Company.) E.P. 295,142. Aug. 9, 1928.

The improvement consists in the method of transferring oil from one part of a distillation unit to another, and to the regulation of the distillation in the system. The oil is transferred from one still to the next by means of a fluid injector arranged in the lower portion of the pipe connecting the stills and having downflow and upflow sections of substantial length. The injector is operated by means of a gaseous or vaporous fluid introduced into the liquid oil. Steam may advantageously be employed as the vaporous fluid so as to obtain simultaneous distillation and transfer of the oil. H. E. T.

194. Fractionating Tower. W. H. Bahlke. U.S.P. 1,702,805. Feb. 19, 1929.

This invention is a further improvement on a previous application, Serial Number 6152, and consists of an improved type of fractionating tower for distilling petroleum oils.

The improvement consists in having a series of parallel rectangular nipples, through which the oil vapours pass. These nipples have overlying rectangular bubble caps, with spaced members overlying the spaces between the nipples. The latter prevent oil being carried upward by the rising vapours. H. E. T.

195. Apparatus for Distillation, Rectification or Evaporation. A. Carpmael, from I. G. Farbenindustrie A.-G. E.P. 299,084. July 20, 1927.

A vapour outlet duct from the distilling vessel is shaped to reverse the direction of flow and is progressively smaller in cross section. The constricted end discharges downward into a reflux tube leading to the bottom of the distilling vessel. G. S.

196. Orifice Mixer. C. L. Erickson. U.S.P. 1,698,432. Jan. 8, 1929.

An orifice mixer, particularly suitable for producing thorough mixing of petroleum distillates, with small quantities of non-miscible reagents such as sulphuric acid, water, alkalies or sodium plumbite solution is described. Diagrams showing the general arrangement of the mixer are given. H. E. T.

197. Refining Oil. C. B. Forward. U.S.P. 1,698,811. Jan. 15, 1929. Appl., Aug. 19, 1919. Renewed March 26, 1927.

A process is claimed of refining oil, and consists of forcing a stream of oil through heating coils, in which superheated steam at 1000° F. is caused to

flow inversely to the flow of oil. The mixed vapours are discharged under pressure into a closed vessel having separate channels of exit for volatilised and liquid products. The volatilised products, together with steam, are reheated in a heater maintained at 700° F. or higher by superheated steam, which is re-used at a lower temperature to heat the oil in its initial flow to the oil heaters. The highly volatilised product is subjected to the washing and cooling effect of water in a closed cylinder under pressure, and the volatilised and liquid contents are separately conveyed from the cylinder for treatment by the customary means. H. S. G.

198. Distillation. International Combustion Engineering Corpn., from W. Runge. E.P. 277,952. Sept. 6, 1927. Conv. U.S.A., Sept. 21, 1926.

The apparatus claimed comprises a hollow member which may be rotated in the still and carry the material being treated as a thin film on its surface, the heating medium being passed through the hollow member. The hollow member may be in the form of a star in cross-section, and may contain a central baffle to direct the medium to the transverse limits of the space. G. S.

199. Dephlegmator. H. S. Mabey. U.S.P. 1,670,118. May 15, 1928.

This invention relates to improvements in dephlegmators for use in the treatment of petroleum oils.

The dephlegmator consists of an external casing with a detachable top, and is fitted with vapour inlet pipes through which oil vapours from the cracking zone are introduced. The detachable top plate is fitted with baffle plates in order to direct the uncondensed vapours to the outlet pipes. The lower portion of the casing contains a number of supports in which tie rods are set. The tops of the tie rods are bent to form rings which furnish a means for removing the rods when the interior of the dephlegmator is to be cleaned. The interior of the dephlegmator consists of a number of pans, shaped in the form of sectors of a circle and so arranged one above the other so that they form a spiral. The pans are perforated with small holes to allow liquid to be drawn through them. At the top of the casing is a raw oil inlet pipe, and at the bottom a draw off pipe for removing raw oil and condensate. The oil vapours on entering the dephlegmator are diverted by the pans through a spiral course through the downwardly dripping oil, and the uncondensed vapours are drawn off through the outlet pipe at the top. Raw oil is admitted through the top of the casing, and in the course of its downward flow causes the hot oil vapours to be effectively dephlegmated and collecting condensate to be carried back to the heating zone. H. E. T.

200. Apparatus for Distilling Oil. H. S. Pettingall. U.S.P. 1,698,507. Jan., 1929.

In the operation of oil stills, particularly when heavy oils are distilled under pressure, deposits of coke are formed on the inner surfaces of the stills. In the present invention, the sediment is removed by a scraping device and pushed towards the outlet. The scraping blades are supported in travelling hollow holders having slots top and bottom through which the blades project, and weights are attached to the blades within the holders. The blades pass over the still bottom in an upright position, and on reaching the end of their travel are inverted and carried through the upper part of the still by means of endless conveyer chains. As the holder and blades are inverted, the weight of the blades draws them into the holder, thus scraping them against the edges of the slots which remove any material adhering to the face of the blades. The holders are guided by a pair of endless tracks parallel

to the chains. The framework supporting the chains, etc., is anchored at one place in the still in such a manner as to permit expansion and contraction. The apparatus is driven by a shaft which projects through the shell of the still at one end. R. P.

201. Mineral Oil Vaporizer. A. E. Pew, Jr. Assr. to Sun Oil Co. U.S.P. 1,700,709. Jan. 29, 1929.

An elongated still chamber is provided internally with an inclined vaporizing pan, having a reticulated cover, the openings of which are in free communication with the vapour space in the chamber. Two pans may be provided and inclined in opposite directions. G. S.

202. Valves for Filling Tanks, Reservoirs and the Like, being Operated by Means of a Float. A. Purser and G. D. Hook. E.P. 299,499. July 7, 1927.

A piston on the low-pressure side of the main valve is arranged to raise the main valve from its seat, the piston being operated by a float actuated auxiliary valve controlling the supply of liquid to the piston from the high pressure side of the main valve. G. S.

203. Process of Utilising Carbonaceous Material. D. Pyzel. U.S.P. 1,699,989. Jan. 22, 1929.

By means of this process heavy carbonaceous material, such as oil sands, oil shales, asphalt and heavy residuum from refining operations, is converted into more valuable products. The apparatus consists of a furnace, condenser and gasometer. The furnace is operated on a principle which bears many resemblances to that of blast furnace practice.

The carbonaceous material is mixed in the vertical retort with inert or non-combustible material in the form of pieces of mineral matter which will stand high temperatures without material disintegration. Steam is injected into the lower portion of the porous mass in the retort to generate water gas, while air is injected above the steam inlet to form a combustion zone. The combustion gases rise to the top of the porous mass in the retort, and assist the distillation of the volatile matter from the carbonaceous material. Part of the inert material is continually removed from the bottom of the retort and then transferred to the top of the retort. H. E. T.

204. Apparatus for Detecting the Presence of Infalmmable Gas. W. M. Thornton. E.P. 298,767. Oct. 11, 1927.

The apparatus is constructed as a miner's electric lamp and comprises two electrically-heated filaments, one for illumination sealed in air and the other exposed to the atmosphere under test. By adjusting a variable resistance until the two filaments glow with equal luminosity, the percentage of inflammable gas present can be ascertained. G. S.

See also Abstract No. 269.

Engineering.

205. Protection of Underground Pipe from Corrosion. E. O. Slater. *Ind. Eng. Chem.*, 1929, **1**, 19-21.

The methods suggested for protecting underground pipes from corrosion are :—

1. Manufacture of corrosion-resistant pipe.

2. Modification of the condition of the soil, such as placing the pipe in Portland cement or lime.

3. The use of protective coatings. This is the method commonly used. The rate of corrosion is determined in a broad way by the character of the soil in which the pipe is laid, and hence any coating to be effective must keep the soil and soil water out of contact with the pipe. The protective coating must be applied to the pipe which has been thoroughly cleaned by sand-blasting.

The protective coatings available for underground pipe lines are :—

1. Bituminous, such as native and residuum asphalts, coal tar pitches and gilsonite. These are applied cold as paint or by dipping or pouring.

2. Reinforcing wrappers of felt, cotton or mineral fibre usually impregnated with bitumen.

3. Metallic coatings.

Asphalts are less brittle than the coal tar pitches, while paints require considerable time to dry, but tend to adhere better than hot applications. Of the fabric wrappers, cotton is the best as it will impregnate easily, and forms a wrapper which does not tend to unwind.

A coating successfully used in Southern California consists of a combination of a paint and reinforcing cotton wrapper.

One product successfully used consists of native bitumens from Trinidad, Bermudah and Utah. The priming coat, called "Penetration," was applied by hand brushing, 1 gallon covering 300 sq. ft. This coat, which dries after two hours, was followed by a heavy coat, called "Electrolysis Proof," 1 gall. of this material covering 160 sq. ft. After a day's drying a second coat of "Electrolysis Proof" is applied. This forms a tough resisting coat of sufficient protection in good soils. For more efficient protection, the above treatment is followed by a spiral wrap of impregnated cotton fabric, followed by two coats of heavy "Electrolysis Proof" coating.

The results are given of a series of electrolysis resistance tests on specially prepared specimens of pipe coated with one coat of Electrolysis Penetration Coat and two coats of Electrolysis Cover Coat, three days being allowed between the coats for drying.

H. E. T.

206. A Full Diesel Engine. *Automobile Engineer*, April, 1928.

A description is given of the M.A.N. Diesel engine as manufactured in America. This consists of a four-cylinder, solid-injection full Diesel unit. The cylinders are 6 in. bore, 8 in. stroke and operate on the four-stroke cycle. Either electric starters or a two-cylinder petrol engine can be used for starting; a manual controlled pressure release is fitted to assist starting. The valves are located vertically in the cylinder heads and are operated by rocker arms and push rods. The inlet valve has an integral deflector on the head to produce the necessary turbulence. All parts are completely enclosed. Cooling is by means of a centrifugal water pump. Force-feed lubrication is by a dual gear pump operating on the dry sump system. The fuel pump is mounted on a bracket integral with the crank case and is driven by the timing gear train; it is a cam-actuated plunger type with a plunger and cut-off valve for each cylinder.

The speed of the engine is controlled by the point of the fuel cut off and the duration of the injection, these two are controlled by hand controls. Comparative performances with petrol engines of the same size are given, thus when both are developing 100 b.h.p. at 700 r.p.m. the Diesel engine consumes 6 gallons per hour, while the petrol engine consumes 10 gallons.

W. N. H.

207. Floating Cover for Oil Tanks or the Like. C. R. Gallagher. E.P. 300,086. April 17, 1928.

A buoyant cover for oil tanks is made of a number of horizontally sector-shaped pontoons arranged to form a circular cover, and provided with upward extended marginal walls to provide water collecting pans. Means may be provided for equalising the water level in the various pans and a discharge pipe arranged in communication with all the pans.

G. S.

208. Rotary Oil Burner. John Scherninger, Jr. U.S.P. 1,695,030, Dec. 11, 1928. Appl. May 10, 1926.

The fire box is provided with a fire deck having a central opening and spaced air ducts extending outwardly and upwardly around the combustion chamber. A rotatable oil distributing device is mounted in the opening and has a restricted peripheral opening for the discharge of atomised oil, causing a thin film of oil to issue over the surface of the fire deck. Means are provided for inducing a current of air partly through the ducts and largely under the film of oil.

G. S.

209. Liquid Preparation for Cleaning Internal Combustion Engines. J. W. Mellors. E.P. 304,923, Feb. 2, 1928.

A preparation for cleaning the interior of an internal combustion engine consists of a mixture of naphthalene and sodium carbonate mixed in about the following proportions with 1 quart of petrol :—

For aeroplane and high speed engines : 7 oz. naphthalene, 2 oz. carbonate.
For 30-100 h.-p. engines : 5 oz. naphthalene, 1 oz. carbonate.
For 30 h.-p. and under engines : 3 oz. naphthalene, 3 oz. carbonate.
For very small power engines : 1 oz. naphthalene, 3½ oz. carbonate.

G. S.

See also Abstracts Nos. 151, 174, and 205.

Cracking.

210. Cracking in the Vapour Phase. S. J. M. Auld. *Petr. Times.* 1929. **523, 524.**

The first part of the article is mainly historical dealing with the work of Young, Dewar, Redwood, Burton, Sabatier, Hall, Rittman and Ramage, and the second part consists of a description of the plant and operation of the Gyro process which has been developed from the Ramage process. The Ramage process came into prominence when liquid phase processes were beginning to supercede the older vapour phase processes, and although it was not completely abandoned, development was practically suspended for some years. The recognition of the anti-knock value of vapour phase spirit, however, has put the process on a different competitive basis as Gyro petrol has, at least, an anti-knock value equivalent to a blend of 50 per cent of benzol in a straight run spirit. The high anti-knock value may be ascribed partly to the aromatic and partly to the unsaturated hydrocarbon content. At present vapour phase cracked spirit is the only anti-knock material obtainable from petroleum in unlimited quantity, and it has the advantage that the anti-knock quality is independent of the source of the raw petroleum.

A diagrammatic flow sheet of the Gyro process explains the method of operation.

As in all vapour phase processes, fuel consumption is high, but there is no re-running and the gas produced is sufficient to provide fuel for all purposes. R. P.

211. Safety Appliance. L. C. Huff and A. G. Bogardus. U.S.P. 1,703,624. Feb. 26, 1929. Appl., July 5, 1923.

As a safety device in a cracking plant, an outlet pipe is provided with means, such as a plate, by which it may be broken at a predetermined pressure. A funnel-shaped member located above the outlet pipe is furnished with a steam line which is closed with a fusible plug. G. S.

212. Means for Treating Oils. C. B. Belknap. U.S.P. 1,704,277. March 5, 1929. Appl. May 7, 1925.

Oil-treating apparatus is provided with means to maintain the liquid at a predetermined pressure and so arranged to prevent vapour generation until the oil has been heated at a predetermined pressure, and thereafter providing a vapour space to receive the vapours generated. G. S.

213. Process for Converting Hydrocarbon Oils. C. P. Dubbs. U.S.P. 1,703,100. Feb. 26, 1929.

The process provides a method of preheating the raw oil by circulating it through a closed tube within a refluxing column. Simultaneously, the vapours are cooled and the heavy fractions are condensed to form the reflux oil. Further, to assist the refluxing action, the overhead distillate is returned to the upper part of the column.

The plant consists of a heating coil, expansion chamber, dephlegmator of special design, a condenser, and gas separator. R. P.

214. Process of Treating Hydrocarbons. G. Egloff. U.S.P. 1,692,203. 1928. Appl. July 19, 1918.

The process consists in heating a heavy oil in the presence of aluminium chloride to a temperature exceeding the melting point of the catalyst, withdrawing and condensing the vapours and continuously removing the mixture of oil and aluminium chloride from the bottom of the still and returning it to the top thereof. H. G. S.

215. Process for Cracking Emulsified Oil. G. Egloff and H. P. Benner. U.S.P. 1,703,103. Feb. 26, 1929.

The invention relates to improvements in apparatus for cracking emulsified oil in which the vapours are subjected to reflux condensing action and the reflux condensate is separately collected whilst steam and lighter vapours are removed, thus preventing any tendency of the heavier fractions to re-emulsify with the condensed water.

The plant consists of the usual heating coil, vapour chamber, and dephlegmator. Thence, the vapours pass to a condenser coil which is maintained at a temperature above the boiling point of water at the pressure used. The steam and vapours which do not condense in the first condenser pass to a second cooling coil at a temperature at which the light oil and water condense and run to a receiver where the water separates. The condensate in the first condenser is passed through a cooler into another receiver.

The vapours which are condensed in the dephlegmator are allowed to flow through a third condenser which is also maintained in temperature above the boiling point of water and any steam or light vapours are conducted into the second cooling coil whilst the heavier condensate, free from water, flows through a cooler to a receiver. R. P.

216. Desulphurization of Mineral Oils. G. Egloff. U.S.P. 1,703,616. Feb. 26, 1929.

This process refers to the removal of sulphur compounds from hydrocarbon oils and particularly to the refining of cracked oils. It consists of distillation of the oil in the presence of fish oil, chinawood oil or other drying oil, with which the sulphur compounds combine. As an example a pressure distillate is mixed with 3 to 5 per cent. of drying oil to a temperature of from 250 to 300° C., depending upon the type of oil to be tested. It is preferable to maintain the mixture at a pressure of from 50 to 200 lbs. during the distillation or digestion. The process may continue for 10 to 24 hours, and the overhead distillate is practically free from sulphur compounds, which remain with the heavy ends and drying oil in the residue. R. P.

217. Process for Cracking Oils. G. Egloff. U.S.P. 1,703,617. Feb. 26, 1929.

In a battery of cracking stills, each unit, consisting of a heating coil, expansion chamber, dephlegmator, condenser, and gas separator, is working independently of the other units, but the gas outlet from each separator is connected to a common main conducting the permanent gases to a compressor which compresses the gas and passes it into a storage receiver. From the receiver, the compressed gas may be pumped back into any of the feed lines to the heating coils and recycled through the plant, along with the mixture of reflux and raw oil. R. P.

218. Treatment of Distillates from Processes of Cracking Petroleum Oils. R. A. Halloran. U.S.P. 1,704,246. March 5, 1929.

Pressure distillate from a cracking plant is condensed and passed through a series of vertical tanks without releasing the pressure. The oil enters the first tank at the bottom, overflows at the top and enters the second tank at the bottom, continuing thus through the series. Either salt or fresh water at ordinary temperature is sprayed into the top of each tank, except in the last, which receives caustic soda solution. The quantity of water used amounts to 2 to 2½ times the quantity of oil in each tank. Thus with four water tanks the water used will be 8 to 10 times the quantity of oil treated. By water treatment a large proportion of the H_2S is removed and the final soda treatment absorbs the remainder along with any acid oils which may be present.

By removal of the H_2S and acid substances present in the oil before further refining takes place, a saving of plant by eliminating corrosion and a saving of sulphuric acid for refining are effected. R. P.

219. Continuous Process for the Refining of Oils. E. T. Hessle. U.S.P. 1,696,913. Dec. 2, 1928.

The conversion of hydrocarbon oils into lower boiling products is the object of the process. The oil to be treated is pumped through a heating coil whilst hydrogen-containing gases are preheated in another coil. The stream of oil and the stream of gas are directed against each other by opposed jets, to produce an oil fog which is passed into a molten alloy of tin and antimony, containing not more than 10 per cent. of antimony. The vapours produced are led into a tower, the base of which contains iron and manganese oxides or other suitable desulphurizer, then pass upwards through iron or steel turnings into a second tower which is divided into three compartments containing iron, copper, and nickel turnings respectively. These turnings act as hydrogenation catalysis and are arranged in order of their respective temperature requirements for efficient action. Condensate runs back from

the tower for recycling and uncondensed gas and vapours pass through a final condenser, to separate the light product. The gases are compressed and returned to the process. R. P.

220. Process for Refining Oil. E. T. Heasle. U.S.P. 1,702,540. Feb. 27, 1929.

The oil to be converted is pumped through a preheating coil and raised to 200° to 350° C. under a pressure of 3 to 8 atmospheres, depending on the character of the raw oil. From the coil the oil passes to a fog producing device consisting of opposed nozzles situated in a small compartment within a reaction chamber. The reaction vessel contains metallic tin with not more than 10 per cent. antimony and the temperature is maintained at 250° to 400° C. To produce the oil fog the oil from the preheater passes to one of the nozzles, whilst hydrocarbon vapours are supplied to the other nozzle. The pressure produced in the small compartment of the reaction vessel forces the fog under the baffle and up through the molten metal thence to the fractionating column. The base of the column is filled with a desulphurizing agent, such as the oxides of iron and manganese to remove sulphur compounds. Above the base, a condensate trap is provided and the upper part of the column filled with packing rings. From the top of the column, the vapours are led into a second fractionating tower thence to the condenser.

In this process the production of unsaturated hydrocarbons is encouraged, whereas in a previous patent (1,696,913) using the same apparatus, hydrogenation was desired. R. P.

221. Process and Apparatus for Converting Hydrocarbons. L. C. Huff. U.S.P. 1,685,488. 1928. Appl. Feb. 19, 1923.

The velocity of flow of oil through tubular heaters is increased by the injection of a superheated inert gas, e.g., steam, in successive sections of the heater. The steam is injected at a higher pressure than that prevailing in the apparatus and more efficient heating of the oil takes place. H. G. S.

222. Apparatus for Removing Carbonaceous Material from Oil-Treating Apparatus. L. C. Huff. U.S.P. 1,703,623. Feb. 26, 1929. Appl. June 15, 1925.

A cracking plant is provided with an enlarged chamber in which carbon is accumulated and in which flexible metal elements are supported so as to be removable. One element extends through the centre of the chamber and a second is located outside the uprights surrounding the central element. G. S.

223. Process for the Hydrogenation of Organic Substances, Especially of the Products of the Distillation of Coals and Petroleum Oils. G. Hugel, M. Paul and M. Boistel. E.P. 286,206. 1928.

Hydrogenation of coal, coal-tars, tar derivatives and mineral oils is effected in the presence of catalysts consisting of the hydrides of the alkali or alkaline-earth metals, the reactions taking place under pressure. The catalysts may be prepared by simple contact with hydrogen under pressure and may be stabilised by the addition of an oily material like tetrahydronaphthalene or substances like the amides of the alkali or alkali-earth metals which are more easily oxidised than the catalyst itself. The latter may also be dispersed on a carrier. As an example, coal-tar pitch is treated with hydrogen at 300° C. and a pressure lower than 100 kilograms in the presence of about 10 per cent. of sodium hydride, the product being a reddish-brown liquid with a green fluorescence. H. G. S.

224. Treatment of Mineral Oils by Heat. W. J. Perelis, Assr. to Shell Development Co. U.S.P. 1,701,477. Feb. 5, 1929. Appl. June 24, 1925.

Heavy heterogeneous oil stock is coverted into gasoline and higher boiling products by separating the light fractions and cracking the less volatile constituents. A heavier fraction of the more volatile cracked products is separated and mixed with a heavier fraction of the lighter fractions of the feed stock, and this mixture is cracked and the products in the gasoline range collected. G. S.

225. Process for Refining Mineral Oils. A. S. Ramage. U.S.P. 1,702,313. Feb. 19, 1929.

The process is a continuous washing method and the plant, as illustrated, consists of three vertical conical-bottomed settling tanks, three mixing chambers set below the level of the settling tanks and the necessary pumps which are synchronised to deliver the proper proportions of oil and acid or soda.

The first mixing cylinder receives oil and acid near its bottom and the stream meets a revolving screw propeller which drives the mixture upwardly against radially arranged vanes and through a perforated diaphragm extending across the mixing chamber below the exit at the top. The intimately mixed liquids pass through a cooler if desired, into a pipe extending downwardly towards the bottom of the first settling tank. This pipe is provided with extending arms to distribute the liquid rising from the bottom of the tube. The settling tank has a conical bottom provided with an outlet valve through which sludge can be drawn continuously or intermittently. The clear oil rises to the top of the settling tank and is allowed to flow through a pipe into which the requisite amount of a caustic soda solution is injected. The mixture enters the second mixing chamber, is agitated and passes into the second settling chamber which is of the same construction as the first. The overflow from the second chamber passes to the third section of the apparatus, where a final treatment with water completes the treatment. R. P.

226. Improved Process for Distilling or Cracking Oils. A. L. Rispler. E.P. 305,263. Feb. 4, 1929.

A cylindrical still rotates within a furnace and the temperature is raised high enough to volatilise at once the volatile portions of the raw oil, whilst the asphaltic portions drop into the still. The still is partly filled with crushing members and iron balls, which roll loosely within the still and pulverise the carbon deposit.

The oil enters at one end of the still through a stationary pipe fitted to the still by a gas-tight stuffing box and gland, and the vapours produced pass out at the other end through a similar fitting.

The still is cleaned by blowing out the powdered carbon with steam.
R. P.

227. Pressure Distillation of Hydrocarbon Oils. F. M. Rogers and E. J. Shaeffer, Assrs. to Standard Oil Co. of Chicago. U.S.P. 1,697,573. Jan. 1, 1929.

A method is claimed of distilling hydrocarbon oil for the formation of gasoline containing distillate and consists of heating a quantity of oil having a boiling point range between 450° and 750° F. under a pressure of 5–6 atm. to conversion temperature and introducing into the vapours evolved, gasoline containing distillate in amount at least equal to that of the evolved product. The distillate introduced should have approximately the characteristics

of the desired product and may conveniently be the product of a former run. The mixed vapours are removed and products of a higher boiling point than desirable are returned to the body of the oil. H. S. G.

228. Device for the Distillation and Depolymerization of Liquid Hydrocarbons. A. A. F. M. Seigle. U.S.P. 1,702,275. Feb. 19, 1929.

This cracking plant consists of a vertical retort provided with a central heating conduit, and a passage for the oil vapours formed by a series of annular chambers surrounding the central flue. These chambers are arranged to cause the vapours to travel alternately in opposite directions round the flue. Heat is supplied at the top of the flue by a flame jet and the products of combustion are drawn downwards by a motor turbine.

The oil to be treated is preheated and supplied to the bottom of the retort. The vapours are heated at successively higher temperatures as they pass from chamber to chamber on their upward course. The working pressure is practically atmospheric and the temperature at top of the retort is about 600° C.

From the retort, the vapours pass successively through expander coolers which consist of a cylindrical outer case and a conical inner chamber. The inner chamber contains perforated plates which support metal chips through which the vapours pass and steam fills the annular space between the chamber and the outer case. In the first expander, the steam temperature is maintained at 250° to 300° C. and in the second at 120° to 130° C. Thus the highly-heated vapours are subjected to two sudden coolings. A condensing apparatus separates the liquid products. R. P.

229. Improvements in Methods and Apparatus for Catalysing Gaseous Reactions. The Silica Gel Corporation. E.P. 304,251. Jan. 14, 1929.

The invention relates to a vapour phase oil cracking process, wherein a finely-divided catalyst, consisting of silica gel having associated with it an active material such as iron oxide capable of promoting the reaction, is fed into the vapour so as to form a suspension of the catalyst in the vapour stream. The vapour and suspended catalyst pass through a reaction zone maintained at the desired temperature, then the catalyst is separated from the cracked vapours, reactivated, and returned to the process.

The process is continuous and should carbon be deposited on the catalyst, this carbon is burned off in the reactivating step, which consists of heating in an oxidising atmosphere.

The plant consists of a closed retort to which the raw oil is fed and in which it is vaporized. The vapours pass to a specially-designed cracking tower at the bottom of which pulverized catalyst is fed into the vapours by a worm conveyor.

The vapours leaving the converter are conducted to a separator, a large conical vessel, thence to the condenser, which is vertically arranged. A water spray washes away any catalyst carried over from the separator into the condenser. The condensate is conveyed to a separator in the form of a Dorr thickener. The catalyst from the thickener is filtered and fed to the activator, whilst the oil product is drawn from the top of the separator R. P.

230. Improvements Relating to the Cracking and Fractionation of Hydrocarbon Mixtures and Apparatus Therefor. Standard Development Co. E.P. 296,919. 1928.

Heavy oils are cracked under pressure and the product separated by means of reflux towers into gas oil, kerosine and gasoline, the vapours of the last

two fractions being directed into about the middle of a tower which is supplied with a controlled reflux. H. G. S.

231. Method of Distilling Hydrocarbons. F. E. Wellman. U.S.P. 1,680,580. Aug. 14, 1928.

This invention relates to the treatment of hydrocarbon oils with special reference to the cracking of petroleum, having for its object the improvement of the usual methods of treating the heavy fractions which are usually separated and refluxed to the cracking still, for the production of an increased yield of gasoline.

The return of the condensate to the cracking still has usually been carried on by direct and simultaneous cooling, and running back of the condensed fractions; but with the present method the return of the condensate is neither direct nor simultaneous with the cooling. The heavy fractions separated out are treated in a supplementary still, and after having their lighter fractions distilled off are returned to the cracking still for re-cracking. The whole operation is a continuous one, fresh oil being introduced by way of the supplementary still. The lighter vapours from both the cracking and supplementary stills are carried though a pipe-line containing a pressure retaining valve to the condenser, where they are finally condensed under atmospheric pressure or under a pressure not exceeding 20 lbs. per square inch. This method of operating a cracking still has the following distinct advantages :—

1. The condensate is heated before being returned to the cracking still, its temperature being thus maintained within the necessary limits to secure the desired fraction in the condenser. This cannot be done with ordinary refluxing.

2. The vaporization due to the supplementary heating takes place out of contact with the vapour stream from the cracking still, and hence adulteration is eliminated.

3. The operation of the supplementary still results in an increased yield of gasoline.

A diagram showing the general arrangement of the plant for carrying out this invention is given. H. E. T.

See also Abstracts Nos. 122, 123, 134, 140, 179, 191, 197, 200 and 259.

Anti-Detonation.

232. Determination of Detonation Ratings. G. C. Brown. *Oil and Gas J.* 1929. **41**. 156.

Curves of per cent. increase in highest useful compression ratio in terms of concentration by volume are given for benzene, toluene, meta-toluidine, para-, ortho- and meta-xylidine, mono methyl aniline and lead-tetraethyl. The values were determined in a 5 in. bore, 7 in. stroke single cylinder engine with a variable compression head.

The power output for the compression range is shown, and superimposed are the curves for each detonating fuel. In some cases the intensity at which knock is measured affects the relative values given to the fuels, but any such differences are only evident if the spirits are of widely different characteristics.

The incidence of detonation occurs at the point where the power begins to fall away from the smooth running curve. If average fuels are tested with a fixed ignition setting, it is found that the ratings are practically identical, whether determined as the compression ratio at which incipient detonation occurs or the ratio at which maximum power is obtained.

The ratio for maximum power varies from about 1·5 to 2·0 ratios above that for incipient detonation, and the loss of mean pressure at this point as compared with the smooth running mixture power curve varies from about 3 per cent. to 7 per cent.

In addition to tests on the variable compression engine, the fuels are correlated by means of power-output spark-advance settings on a fixed ratio engine, 2½ in. bore and 4 in. stroke. The agreement between the two engines is reasonably good. The method of testing by means of ignition advance is discussed in detail, and it is concluded that the point of incipient detonation is a satisfactory guide to rating, but that the maximum power output point is not. Details are given of a bomb in use for the investigation of flame travel and pressure wave effects. R. S.

233. Explosions in Closed Vessels: The Correlation of Pressure Development with Flame Movement. O. C. de C. Ellis and R. N. Wheeler. *Fuel*. 1928. 4.

The apparatus used consisted of a cubical bomb of phosphor bronze, 15 cm. side, in which a central electrode 2 mm. gap was situated. One narrow quartz window ran horizontally across the middle of one side while another ran diagonally across the opposite side. Connections were made so that the vessel could be exhausted and charged with gas, and a pressure gauge of the diaphragm, tilting mirror type, recorded the pressures generated in the vessel. By means of two specially shaped pieces of phosphor bronze, the bomb could be made spherical 14·8 cm. diameter, leaving a slit window across a diameter. A photographic film mounted on a rotating drum in front of the window served to record flame front movements, after-glow phenomena, the pressure record as indicated by the gauge, the instant of the passage of the sparks and units of time.

Mixtures of carbon monoxide and air in varying proportions were charged to the spherical bomb; it was found that with all mixtures, except those in which the flame front travelled very slowly (*i.e.*, excepting those mixtures containing less than 25 per cent. or more than 65 per cent. of carbon monoxide), the moment of maximum pressure rise coincides within less than half a milli-second, with the arrival of the flame front at the boundary of the vessel. When the flame front travels slowly by means of convection it reaches the top of the vessel before it reaches the bottom.

With a cubical bomb and similar explosive mixtures, pressure continues to rise slowly after the flame-front has first touched the walls of the vessel. The moment of attainment of maximum pressure does not synchronise with the moments of complete inflammation but is antecedent to it; a typical record is as follows:—

45·2% CO.—Attainment of maximum pressure	..	5·70	millisecs.
Flame reaches sides of cube in	..	3·9	,,
,, ,, 2 side corners in	..	4·98	,,
,, ,, top corners in	..	6·13	,,
,, ,, bottom corners in	..	6·13	,,

This result is due to the fact that while the flame front is squeezing into the corners, there is considerable cooling of the gases burnt earlier in the explosion at the faces of the cube. W. N. H.

234. Flame Movement in Gaseous Explosive Mixtures. O. C. de Ellis. *Fuel.* 1928. **12.**

Part IX.—The effect of convectional movement in gaseous explosive mixtures near the limit of inflammability on the nature of the flame produced by ignition is considered in detail. Various interesting photographs are published illustrating this effect in explosive mixtures consisting of (*a*) carbon monoxide and oxygen and (*b*) carbon disulphide and air. H. E. T.

235. The Inflammation of Mixtures of the Olefines and Air in a Closed Spherical Vessel. G. B. Maxwell and R. V. Wheeler. *J. Chem. Soc.* 1929. 245.

Experiments have been carried out on the inflammation of mixtures of olefines and air, on lines parallel to those already reported for the paraffins (*J. Chem. Soc.*, 1927, 2069). The olefines used in these experiments were ethylene, propylene and butylene. The mixtures, which were initially at room temperature and atmospheric pressure, were ignited by means of a secondary discharge at the centre of a closed spherical vessel of bronze. The capacity of the spherical vessel was four litres, and it had its interior surface highly polished. Records were made of the pressures developed and their rates of development.

Graphical comparisons are made between the variation of the maximum pressure, both calculated and observed, and the compositions of the mixtures. A marked displacement of the observed maximum pressure mixture from the theoretical mixture for complete combustion was noted. This result had previously been observed in the paraffin series. These displacements are attributed to the effect of dissociation, since the calculated values for which an allowance is made for dissociation are similarly displaced.

The "lost pressures" are greater than those previously recorded for the paraffins, for which the highest value was 9·8 per cent. with methane. With the olefines examined, the highest loss of pressure occurred with propylene, the loss being 16·5 per cent. H. E. T.

236. Metallic Colloids and Knock Suppression. H. L. Olin and W. J. Jebens. *Ind. Eng. Chem.* 1929. **1.** 43.

Reference is made to an earlier paper (Vol. 18, p. 1316, 1926), in which were given results of tests showing that detonation was reduced by the addition of tetraethyl lead and nickel carbonyl to the fuel, but that no appreciable effects followed the addition of specially prepared lead and nickel sols.

Since Sims and Mardles, working on the same problem, had obtained diametrically opposite results, it was thought advisable to review the earlier work.

The tests quoted give the per cent. increase of H.U.C.R. above straight gasoline for the addition of nickel carbonyl and ethyl lead, and also the values for the sols prepared by heating the dopes at various temperatures, including those used by Sims and Mardles.

The authors conclude that there is a minimum temperature below which decomposition is incomplete and at or above which the sols obtained are practically ineffective. It is suggested that in the nascent state at the high temperatures of the combustion chamber, the effects of metallic colloids may be radically different from those shown under the conditions used.

R. S.

237. The History and Development of Anti-Detonating Agents for Motor Fuels. H. S. Tegner. Paper read before the Institute of Fuel. April 18, 1928.

The author quotes the following list of anti-knock materials:—

	Relative efficiency by volume.
Aniline	11·45
Benzene	1·0
Toluene	1·093
Xylene	1·20
Alcohol	1·85
Ethyl Iodide	13·87
Xylidine	12·03
Toluidine	11·86
Tetraethyl Tin	20·4
Diethyl Selenide	62·5
Diethyl Telluride	250·0
Iron carbonyl	250·0
Nickel carbonyl	277·0
Tetraethyl lead	528

Residual deposits in the engine of an objectionable nature are left by iron carbonyl and nickel carbonyl. Ethylene dibromide was found to be the most suitable substance to be used with tetraethyl lead in order to remove the lead in the form of the bromide through the exhaust valve.

Tetraethyl lead is at present manufactured by heating lead-sodium with ethyl chloride in an autoclave and distilling off the lead tetraethyl formed. Ethyl fluid is 54·5 tetraethyl lead, 36·4 per cent. ethylene dibromide, 9 per cent. halowax oil.

An account of the history and the progress of ethyl fluid in the U.S.A. is given. W. N. H.

238. The Electrical Characteristics of Spark Gaps and Sparking Plugs. E. A. Watson. Paper read before the Society of Automobile Engineers, 1928.

The majority of the paper dealt with the engineering and purely physical aspect of the subject. It was mentioned that a sharp decrease was observed in the spark gap voltage at the onset of pinking, due to the increased ionisation present in the explosive mixture residual from the previous pinking explosion. The author suggests the use of a suitable instrument for measuring this voltage as an automatic pinking indicator. W. N. H.

239. A Method for Preventing the Knocking of Motor Fuel. Gebrüder Avenarius (Berlin) and H. D. Elkington. E.P. 277,326. 1927.

A motor fuel consisting entirely, or almost entirely, of hydrocarbons normally liable to knock is improved by the addition of from 0·3 to 0·5 per cent. of an ester or esters of a fatty acid of high molecular weight or oils consisting largely of glycerol esters such as olive oil. R. S.

See also Abstract No. 210.

Oil Field Technology.

240. Crooked Holes Cost Industry Vast Sum. J. L. Dwyer. *Oil and Gas J.* 1928. **14**. 29.

In the Greater Seminole area there appears to be much evidence to indicate that a large number of the wells sunk have diverged very considerably from

the vertical. A crooked hole may often be blamed for twisting off drill pipe when no other definite cause for breakage is evident, and the cost of drilling a crooked well may be much higher than a vertical well due to this cause alone.

When the site for drilling operations is decided, a point is chosen where there are the greatest chances of striking a rich pool. If the well diverges greatly from the vertical, the producing sand may be penetrated at a considerable distance from the desired spot, and although the chances are probably equal on striking a rich or poor zone, the well has not achieved its object in penetrating the point which had been decided upon as being desirable. One case is recorded of a well drilled by rotary tools to 3500 ft., where the drill was found to be working at an angle of 20° from the vertical. This well was extended by cable tools to a depth of 5100 ft., at which depth the angle had increased to 33°. A survey of the hole showed that the bottom was almost 1400 ft. away from a point directly below the centre of the derrick floor.

It is suggested that the wild divergence in depths at which the Wilcox sand is reached in different wells in the Seminole Field may be due in part to the fact that some holes are considerably more crooked than others, and in the case of wells drilled close together, this seems the only reasonable explanation. E. R. B. S.

241. Determining Deep Well Pump Action. F. W. Lake. *Oil and Gas J.* 1928. **27** (30). 34, 100, 102.

For given pumping conditions, two factors are observable: (1) the motion of the polished rod in relation to a fixed point, and (2) the load changes during the motion. The motion of the plunger in respect to the working barrel must be derived from the other two.

In order to measure the polished rod stroke and load and to calculate the plunger movement, an electric dynamotograph was designed.

The load on the polished rod is measured and recorded by a telemeter on a calibrated rod supporting one-half of the load. The polished rod stroke is measured by a vertical resistance wire, on which slides a contact attached to the polished rod. The plunger stroke is equal to the polished rod stroke less the load multiplied by a constant, and this is calculated automatically and the three readings recorded on charts. The circuit of the dynamotograph is given, and also the results of a series of tests. As anticipated, volumetric efficiency and plunger velocity are related. As the velocity increases to about 1000 in. per minute, the efficiency increases rapidly to a maximum of nearly 70 per cent., but with further increase in velocity the efficiency decreases.

For the lower values of velocity, increases in production are more rapid than at the higher ones.

The percentage of elongation of plunger stroke over rod stroke increases from 90 to 125 per cent. between velocities of 500 and 1800 in. per minute. This permits of rod fatigue being estimated with regard to plunger velocity. G. S.

242. Oil Recovery by Use of Heated Gas. B. E. Lindsly. *Oil and Gas J.* 1928. **27** (31). 27, 75, 76.

The application of hot gas under pressure to oil sands will cause the light fractions of the oil to vaporise and the heavier to "crack," and the oil to flow more readily toward producing wells. The dissolved gas and gasoline will move first, and on coming into contact with sands away from the source of heat and pressure, will condense and dissolve in the original oil, lowering

its viscosity and causing it to flow more readily. The operation will be progressive and vaporise heavier fractions as the heat increases.

The process involves the combustion of methane and air under pressure and forcing the products, with excess gas or air, into the sands under pressure.

The proposed method of injecting the hot gases is by sinking a shaft and driving galleries in four directions at least 50 ft. into the sand. The shaft is plugged for at least 50 ft. above the top of the sand, pipes passing through the plug to convey the combustible mixture and excess gas to the combustion chambers in the oil sand. An electric ignition device, which can be lowered and removed, is to be provided.

Assuming that 19,000,000 cub. ft. of gas are to be compressed daily, the cost of compressors, including upkeep, would be $760, and for exploiting sands at 1000 to 1200 ft. the costs of the shaft and drifts would be about $120 per day over three years. Fuel costs would be $172 per day if oil at $1 per barrel were used, and $168 per day if gas at 15 cents. per 1000 cub. ft. were employed. G. S.

243. Gas Pressure and the Recovery of Oil. R. Van A. Mills. *Oil and Gas J.* 1928. **27** (31). 28, 75.

The pressure gradient is generally less for shallow beds than for those deeper, becoming less again at about depths of 4000 ft. The rule that initial rock pressure is equal to the pressure of a column of water standing vertically to the same depth should only be used with discretion. This pressure equals 43·40 lb. per 100 ft., but is too high for the beds below 1000 ft. and over 4000 ft. deep in Oklahoma. The same remark applies to the Rocky Mountain fields. G. S.

244. Effect of Shutting In Offset Wells. C. M. Nickerson. *Oil and Gas J.* 1929. **27** (40). 87, 91, 95, 96, 98.

In the Californian oilfields that are past the flush production stage, subsurface conditions show the upper portion of the zone to contain mainly wet gas, the oil being in the lower portions.

If wells in the flush stage are shut-in, offset wells have a larger drainage area, their production increasing and gas-oil ratios remaining constant or decreasing.

The shutting-in of pumping wells with a low fluid level, results in an increase of oil production in adjoining wells, and an increase in gas and gasoline and in gas-oil ratios to the maximum of inefficiency.

Where gravity is the principal force in moving the oil, little change is caused by shutting-in.

The increase in gas production in wells which are declining in oil production is due to gas migrating from around the shut-in well and by-passing the oil, the net result being to curtail ultimate oil recovery. Accurate records of oil and gas production will quickly indicate the necessity for remedial action.

The use of mud fluid in the shut-in well would tend to prevent vertical gas migration, and in some cases, the maintenance of back pressure on the producing well would prevent production of excess gas. G. S.

245. Process of Effecting Reaction Between Liquids Tending to Form Tight Emulsions. F. H. McBerty. U.S.P. 1,698,270. Jan. 8, 1929.

The object of this invention is to secure the desired reaction between a contaminated oil and a counter-colloid which is added to the oil in order to remove the impurities. This can be accomplished by mixing the liquids without excessive agitation so that excessive dispersion of the internal

phase is prevented, then separating the mixture into its components and then re-mixing the components. This operation is continued until the desired maximum reaction between the components has been effected. The process is applicable to all emulsions, and is useful in all cases where reagents are added to liquids to produce emulsions whose subsequent separation is difficult. Various applications of the process are given, together with a diagram of the apparatus used. H. E. T.

246. Method of Concreting Shafts, Bore Holes and the Like. T. Blandford and A. Gee. E.P. 301,222. Dec. 21, 1927.

This method relates particularly to the formation of a plug at the bottom of a flooded shaft, and consists of depositing a layer of cement at the bottom and allowing large size materials to drop and set therein. G. S.

247. Recovering Oils from Emulsions. S. W. Cole. U.S.P. 1,700,627. Jan. 29, 1929. Appl., Dec. 17, 1924.

Crude petroleum emulsion is mixed with metallic sodium and calcium carbide, causing a chemical reaction between the water and the sodium and carbide, the water and oil separating into layers. G. S.

248. Apparatus for Earth and Rock Drilling. A. M. Gildersleeve and C. L. Dean. E.P. 280,532, Nov. 3, 1927. U.S.P., Nov. 10, 1926.

A drill bit is described for deep well drilling, comprising a combination of rotary bit body, several exteriorly located rotatable cutters with cutting edges of frusto-conical shape mounted on the lower end of the bit body in fixed relation to each other and inclined so that the cutting edges engage the material to be cut at angles substantially less than 90°. Each rotatable cutter is mounted on a shaft removably located in an opening in the bottom of the rotatable bit body, and has ball bearings to carry the actual thrust on the cutter. An annular groove on the shaft is provided for locking it in place by means of a movable metal piece. The cutters are disposed at such an angle, that as the cutting edge wears away it maintains an effective cutting angle with the conical surface. The cutters are arranged to cut a series of relatively narrow concentric paths of different diameter which collectively cover the bottom of the hole, one of the cutters being arranged to cut a path as closely as possible to the centre of the hole and having its axis inclined outwardly so as to cause the inner portion of its cutting edge to engage the material. The next outer cutter has its axis inclined inwardly to avoid interference with the axis of the inner cutter, a .d the cutters beyond the second have their axes inclined outwardly so as to cause the outer portions of their cutting edges to engage the material. H. S. G.

249. Two-Piece Drum for Rotary Drawworks for Well Drilling Plant. National Supply Co. E.P. 291,049, April 10, 1928. Conv. (U.S.A.), April 25, 1927.

A drum for rotary draw-works consists of two sections united at about the mid cross section of the spool. Each section is provided with an annular shoulder at the inner end, the shoulders projecting inwards and engaging opposite ends of a ring, thus forming an annular groove for welding material. G. S.

250. Elevators for Raising and Lowering Well Casing and the Like. F. J. Young and J. A. Dunn, assrs. to National Supply Co. E.P. 287,889, Sept. 14, 1927. Conv. (U.S.A.), March 29, 1927.

A mechanically operated slip type of casing elevator, comprising a body with a tapered bore in which a number of tapered segment slips are mounted. Means are provided for simultaneously raising and lowering the slips and for moving them radially during raising and lowering. G. S.

251. Deep Well Drilling Apparatus. C. E. Reed. E.P. 296,779, Aug. 22, 1928. Conv. (U.S.A.), Sept. 9, 1927.

A drill having roller cutters recessed to one side of the vertical axis has a vertical wall defining the inner side of each recess, and an upwardly and inwardly inclined wall defining the outer side of the recess. A socket in the drill head communicates with the recess and extends parallel with the axis of the drill. The roller cutter unit has a shank seated in the socket, and a spindle extending into the recess at right angles to the inclined wall. The roller cutter is mounted on the spindle, and the vertical wall serves as a support for the cutter unit. G. S.

See also Abstracts Nos. 125, 128, and 252.

Geology.

252. New Calibrating Device Aids Work with the Magnetometer. L. Spraragen. *Oil and Gas J.* 1929, **27** (40), 118.

In a previous article (see Abstract No. 106) a coil for determining the scale constant for a magnetometer was described, and a general demand for a portable calibrating coil has since been indicated.

The device described consists of a calibrated coil which rests on the magnetometer; a panel carrying an ammeter, potentiometer, resistances and a switch; two dry batteries and a connecting cord. All the parts are fitted in a carrying case 10 in. × 12 in. × 4½ in. The purpose of the apparatus is to set up a magnetic field of known strength and to note the deflection of the magnetometer scale when in this field.

The magnetometer is set up and oriented in respect to the magnetic meridian and the moving system adjusted approximately central by means of the auxiliary magnets. The coil is then placed in position and the current adjusted to one milliampere, the scale deflection for this and for zero current being noted, the difference representing the net deflection for 1 milliampere. This process is repeated up to 10 milliamperes, and the net deflections plotted against the current value and the average deflection per milliampere obtained. From the gamma value per milliampere in the coil and the magnetometer scale deflection per milliampere, it is easy to determine the gamma per scale division. G. S.

253. The Conversion of Fatty Acids During Geological Periods of Time. G. Stadnikow and A. Weizmann. *Brennstoff-Chem.* 1929. X. 61–63.

Engler's theory on the occurrence of petroleum is discussed in this article, and researches on certain Siberian boghead coals are described and numerical results given.

According to Engler's theory that petroleum arises from animal remains, the fatty acids undergo conversion into petroleum hydrocarbons. The

gradual conversion of the carboxylic acids formed into hydrocarbons is termed "bituminisation process."

Stadnikow and Weizmann carried out researches on Siberian boghead coals and were able to show that the coals had arisen from the polymerisation and anhydration products of fatty acids. 800 gms. of finely pulverised coal of the following composition were extracted repeatedly with benzol and chloroform in an apparatus at Soxhlet.

Sucho-Kujataki boghead coal :—

Moisture	.. ..	.. ..	.. ..	4·13%
Ash	.. ..	.. ..	.. ..	7·78%
S	0·76%	referred to	H_2	8·85%
N_2	0·79%	pure coal	O_2	12·78%
C	76·82%		Calorific value	7902

The residue was then heated with 5 per cent. alcoholic caustic soda solution for 50 hours, and after the brownish-black solution had been decanted, the residue was boiled for another 50 hours with fresh alcoholic lye. A similar treatment was carried out with a 5 per cent. amyl alcoholic lye. The residue was washed with ethyl alcohol and then with water. Each extract was tested.

After evaporating off the benzol from the benzol extract, a dark solid remained, similar to crude ozokerite or butiminous peat. This was saponified with alcoholic soda lye and the soap obtained extracted with benzol. The resinous soap was then freed by decomposition with dilute sulphuric acid when semi-solid acids with a characteristic odour of fatty acids were obtained. Lower monovalent saturated and unsaturated fatty acids were shown to be present but in the benzol extract of lignite and peat higher fatty acids were present.

The chloroform extract was not examined.

The extracts with ethyl alcohol soda lye, amyl alcohol soda lye, the residue remaining after the final alcohol wash, and the final water wash showed the presence of caprylic acid, valeric acid (formed by oxidation of amyl alcohol), and butyric acid.

The results give a further experimental confirmation that the Siberian boghead coals are products of intensive polymerisation and anhydration of fatty acids, *i.e.* caproic, enanthic, caprylic acids, saturated and unsaturated acids with 8–9 carbon atoms in the molecule, acids with 9–10 carbon atoms, 10–13 C atoms, and finally high molecular acids, the solid crystalline methyl ester having a melting point of 59–60° and a molecular weight of 347. The latter acids have 22 carbon atoms in the molecule. W. S. C.

See also Abstracts Nos. 115, 116, and 118.

Coal and Shale.

254. Composition of Tar from Low-Temperature Carbonisation of Utah Coal. I. R. L. Brown and B. F. Branting. *Ind. and Eng. Chem.* 1928, **20**, 392–6.

The paper is a continuation of the work of Brown and Cooper (*Ind. and Eng. Chem.*, 1927, **19**, 1927: J. 1927, 115A) and deals with the composition of the aqueous distillate and the medium tar oil produced by the carbonisation of a Mesa Verde (Utah) coal in a continuous vertical retort with steam at a maximum temperature of about 700° C. The quantity of steam employed was 1·5 kilograms per kilogram of coal, the aqueous distillate produced being

1·56 kilograms per kilogram of coal. A 10-litre sample of this aqueous condensate was extracted with ether and the extract washed in succession with sodium carbonate, caustic soda and dilute hydrochloric acid to recover respectively the carboxylic acids, phenols and bases and to leave a residue of neutral oil. These, taken in order, were found to be present to the extent of 3·9, 23·0, 0·824 and 1·0 grams per 10 litres of sample. The carboxylic acids were of a tarry, gummy type, and apparently of a complex nature, whilst the neutral oil was a brown viscous liquid which began to crack at about 200° C. The phenols were fractionated at atmospheric pressure up to 290° C. and than at 5 m.m. pressure to 300° C. The following compounds were detected, viz. phenol, o-, m- and p-cresol, catechol, xylenols (?) and resinous bodies which were not examined. The bases consisted of 83 per cent. ammonia and 17 per cent. pyridines and pyridine homologues.

The medium oil or tar portion 1 constituted 3·91 per cent. of the original coal or 28·85 per cent. (by weight) of the total tar. Its specific gravity at 20° C. was 0·9337. It was resolved by the method used in the treatment of the aqueous distillate into matter insoluble in ether (1·0 per cent. of tar 1), carboxylic acids (0·25 per cent.), liquid phenols (21·0 per cent.), resinous phenols (2·4 per cent.), bases (1·7 per cent.) and neutral oils (66·0 per cent.), the working loss being 7·65 per cent. The ether insoluble matter was mainly coal-dust and resins and the carboxylic acids were too small in quantity to examine. The bases gave negative tests for primary and secondary amines and were considered to be rich in dimethylpyridine and higher homologues. The phenols were found to contain 30 per cent. of solid and non-distillable substances and 50 per cent. of volatile bodies, the remainder being made up of ether, water, oil, wax, and fractionation and other working losses. There were identified phenol, the cresols, 1·2·4 and 1·3·5 xylenol and catechol.

Of the neutral oil, 8 per cent. distilled to 140° C. and 65 per cent. between 140° and 275° C. The remainder consisted of heavy oil 12 per cent., paraffin wax 10 per cent. and resin 5 per cent. Benzene, naphthalene, indene and styrene were absent. H. G. S.

255. Apparatus for the Distillation of Fuels at Low Temperature. A. Abrassart. E.P. 302,875. Jan. 12, 1928. Belg., Jan. 12, 1928.

A retort is describd for the low temperature carbonisation of fuels previously partially distilled at low temperatures in another retort and thereafter formed into blocks with a binding material. The blocks are displaced continuously by means of a flexible movable conveyor composed of a metallic lattice. The actuating members of the conveyor are located outside the retort and the working strand enters and issues from the retort through a bath of sealing material, such as sand or liquid. H. S. G.

256. Distillation of Oils, Coal Tar and Other Liquids. The Gas Light and Coke Co. W. G. Adam and F. M. Potter. E.P.303,038. Aug. 27, 1927.

Coal-tar, after preheating and dehydrating in condensing coils, is passed through two coils in series, leaving the first at a temperature of 225–240° C., and leaving the second at a temperature of 260–300° C. After leaving the first coil it is expanded in a chamber which forms part of a fractionating column and reheating still, operated to give a distillate of high naphtha content and a condensate oil of high naphthalene and phenol content. The residue passes into a second similar chamber with fractionating column and reheating still designed to give a carbolic oil fraction of high naphthalene and cresylic acid content and a creosote oil fraction. The distillates from

the fractionating columns are condensed in coils or other cooling devices which are cooled by the coal-tar before entering the first coil, being thus preheated and deprived of water and light oils which are condensed for recovery of benzene hydrocarbons. The pitch, leaving the second expansion chamber, is caused to flow down scrubbing towers meeting an ascending current of superheated steam which removes all or part of the anthracene oil, leaving a pitch of any desired consistency. H. S. G.

257. Hydrogenising Tar Oil. R. Bernhard and W. Demann. U.S.P. 1,691,221. 1928. Appl. June 25, 1925.

Tar oils are hydrogenated at atmospheric or slightly elevated pressures, *e.g.*, two atmospheres, by means of a current of dried coke-oven gas, a colloidal nickel catalyst being suspended in the oil. Highly-sulphurous tars must first be desulphurised by treatment with, for example, bog iron ore.
H. G. S.

258. Rotary Retort. T. A. W. Dwyer. U.S.P. 1,703,418. Feb. 26, 1929. Appl. Aug. 15, 1924.

A horizontal retort comprises a hollow casing in the shape of a frustum of a pyramid, externally heated, and is arranged to be rotated in a horizontal position. Means are provided for feeding the raw material and the products are drawn off from the upper end of the retort. Alternatively, the retort may have a polygonal cross-section as regards its inner walls. G. S.

259. Improvements in Supplying Heat in High Pressure Reactions. I. G. Farbenind, A.-G. E.P. 275,248. 1928.

The heat required to complete reactions under high pressure, *e.g.* the destructive hydrogenation of carbonaceous materials, is supplied, not through the pressure-resistant walls of the plant, but wholly or partially by pre-heated gases which may or may not take part in the reaction. The heat may be provided by a device, *e.g.* an electrical heater, so arranged as to be out of contact with materials which would be decomposed by, or would react with, the device; or, alternatively, when working with hydrogen, oxygen may be added to burn part of the hydrogen so as to heat the remaining gas to the desired temperature. H. G. S.

260. Manufacture and Production of Liquid and Other Hydrocarbons and Derivatives Thereof by the Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind, A.-G. E.P. 293,719. Aug. 8, 1927. Conv. (Germany) Aug. 7, 1926.

Carbonaceous materials are destructively hydrogenated in the presence of catalysts consisting of cobalt or cobalt compounds together with up to 10 per cent. of metals of the sixth periodic group or their compounds. The process may be carried out at pressures of at least 50 atmospheres.
G. S.

261. Production of Liquid Hydrocarbons by Carbonisation and Hydrogenation of Carbonaceous Materials. J. Y. Johnson, from I. G. Farbenind, A.-G. E.P. 304,343. Oct. 17, 1927.

In the heat treatment of carbonaceous materials in the presence of gases or vapours, the latter are maintained at a high rate of flow by circulation, the reaction products being separated by branching off a portion of the gases.
G. S.

262. Process of Preparing Hydrocarbons. F. A. Howard. U.S.P. 1,702,899. Feb. 19, 1929.

Low-grade coal is ground to a fineness of 100–200 mesh in a tube mill in the presence of sufficient water to form a freely-mobile slurry, which is levigated by imparting to it, in a suitable vessel, a slight upward velocity sufficient to float the coal particles while permitting ash, sulphide, etc., to settle. The semi-purified material is mixed with say 25 to 50 per cent. of hydrocarbon oil. The oil associates with the coal particles and forms a plastic mass which floats on the water and may be separated, leaving the impurities in the water layer. Additional water may be added at this stage to improve the separation. The degree of plasticity or fluidity of the product depends on the amount of oil added.

The carbonaceous mixture is subjected to the action of hydrogen under a pressure of from 50 to 200 atmospheres and at temperatures from 370° to 540° C. but the temperatures will depend on the materials to be treated.

After several hours treatment the products are removed from the pressure vessel and used as fuel or may be subjected to distillation, yielding light and heavy products which are mainly saturated in character. R. P.

263. Treatment of Coal Distillation Gases. Imperial Chemical Industries, Ltd. K. Gordon and J. Hughes. E.P. 304,345. Oct. 17, 1927.

Coke-oven and other gases are scrubbed under pressure in three stages: with aqueous ammonia to remove carbon dioxide and sulphur compounds; with ammonical cuprous solution to remove carbon monoxide and unsaturateds; and with kerosine to remove methane, etc. The first two washes may be carried out at lower pressures than the third, and the absorbed gases may be recovered and used. G. S.

264. Improvements in the Hydrogenation of Coal and Like Carbonaceous Material. C. H. Lander, F. S. Sinnatt and J. G. King. E.P. 302,629. 1928.

In the process of hydrogenating carbonaceous materials under pressure, a portion of the gaseous products is withdrawn through a by-pass, subjected to treatment in order to reduce its content of sulphur compounds and other impurities, and then returned at some determined point into the apparatus. H. S. G.

265. Production of Liquid Products from Coal and Other Solid Carbonaceous Material. M. Melamid. E.P. 285,879. Feb. 24, 1928. Ger. Feb. 24, 1927.

Finely-pulverised coal is mixed with a finely-divided metal catalyst and injected into an autoclave under pressure by means of hydrogen or hydrogen containing gas. Hydrogenation is carried out under high pressure at an elevated temperature for the production of liquid products. H. S. G.

266. Use of Pulverised Coal or Liquid Fuel as Fuel for Steam Generators and the Like. Arthur Musker. E.P. 302,255. Aug. 11, 1927.

The combustion chamber for powered coal or gaseous fuel is a separate vertical chamber, into which the fuel is introduced at the upper end and ignited. The flame of burning fuel is delivered from a lateral lower level to the furnace or flue-tubes, where the chamber consists of annular or spaced metal plates in which the air for combustion is heated before being supplied to the burner to which the fuel is introduced in the chamber and to different points in the chamber. G. S.

267. Coating or Impregnation of Granular Pulverulent or Powdered Materials with Liquid Compositions and Manufacture of Briquettes. H. Nielsen and B. Laing. E.P. 304,812. Oct. 26, 1927.

A powered material to be coated is allowed to fall in a finely-divided stream through a chamber and in contra-flow to an atomised stream of the coating composition. The temperature at the lower portion of the chamber is such as to volatilise any surplus coating material. The coated material is passed to a mixing device and then on to a briquetting machine. G. S.

268. Process for Resolving Emulsions of Tar or Tar Oils. H. W. Robinson and D. W. Parkes. U.S.P. 1,696,057. Dec. 18, 1928.

This invention relates to the recovery of water-gas tar or tar oils from aqueous emulsions or suspensions containing these substances. The term "tar oils" includes distillates of every kind of tar except those containing large quantities of pitch.

The process consists of adding to the emulsion a solid hydrocarbon which is soluble in tar and oil and has a melting point below 100° C., the emulsion being first rendered faintly acid by the addition of a mineral acid. Crude or refined naphthalene is recommended as a convenient hydrocarbon.

In cases where the emulsion already contains the hydrocarbon in the order of 15 to 20 per cent. of the weight of the oil in the emulsion, it is found sufficient to heat the mixture until the hydrocarbon is completely dissolved and then cool with rapid stirring.

The acidification of the emulsion may be accomplished by saturation with carbon dioxide, sulphur dioxide or flue gases.

Acenaphthene, phenanthrene and paraffin wax also serve to effect resolutions of emulsions containing tar oils but are not considered as efficient or desirable as naphthalene. W. L.

269. Distillation of Tar, Oil and Other Materials. Walter Runge, Assr. to International Combustion Engineering Corporation. E.P. 295,945. Sept. 6, 1927. Conv. (United States) Sept. 21, 1926.

Where oils and similar materials are distilled by heat supplied by a hot medium, the temperature of the medium is subsequently reduced and controlled by introducing a suitable proportion of cool air during its passage to the condenser. The admission of cool air may be regulated by a thermostat and the medium may pass through a series of condensers, before entering each of which cool air is admitted in the correct proportions for the condensation of the desired fractions. G. S.

270. Process for the Extraction of Hydrocarbon Oils from Coal. J. Trautmann. E.P. 272,526. Dec. 10, 1928.

Gas and vapour from a coal distillation retort whilst still hot are passed to a hydrogenating apparatus.

Water-gas is generated from the coke produced in the process, and mixed with the vapours. Zinc, tin, nickel or other catalysts may be added in powder or vapour form after hydrogen sulphide or other sulphur compounds have been removed from the vapours and before they pass into the reaction chamber.

The plant specified consists of a single apparatus wherein the gas producer is situated beneath the retort. The gas is passed directly into the retort and the reaction chamber is connected structurally to the retort. R. P.

271. Hydrocarbons from Coal and Water. A. Uhlmann. E.P. 283,177; Jan. 5, 1928. Conv. (Germany) Jan. 6, 1927.

A mixture of coal and water is subjected to the influence of a high-frequency field, which may be produced by pulsating direct current. G. S.

272. Improvements in or Relating to Manufacturing Hydrocarbons. J. A. A. Zacon. E.P. 301,949. 1928.

The raw material, *e.g.* coal, to be hydrogenated is pulverised, freed from ash and amalgamated with oil and then injected, together with water, into a vessel in which the pressure is maintained at about 150 atm. and the temperature at 300° C. The materials in the plant are subjected to the action of an electric arc or spark whilst catalysts may also be employed. An apparatus suitable for the purpose is described. H. G. S.

273. Dehydrating Tar. A. W. Warner. U.S.P. 1,689,309. Oct. 30, 1928.

A comparatively dehydrated mixture of the liquid is circulated through the apparatus and, at a point between the separator and the pump, a limited quantity of the mixture to be dehydrated is added so as to reduce the temperature to a point below that at which frothing will occur in the pump. The temperature of the mixture is then raised so that the water passes freely into vapour, and the vapour drawn off from the separator. Before the addition of fresh liquid for dehydration, a quantity of dehydrated liquid is drawn off equal to that to be added.

The apparatus comprises a separator, a heater with a conduit into the top of the separator, a mixing tank below and connected to the separator and a pump in the conduit between the mixing tank and the heater. Means are provided for feeding the mixture to be treated and for withdrawing water vapour and dehydrated material. G. S.

See also Abstracts Nos. 203 and 223.

ABSTRACTS.

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Abstractors.—S. W. COOKE, B.Sc., R. W. L. CLARKE, B.Sc., W. S. CLARKE, B.Sc., H. S. GARLICK, B.Sc., W. LITTLEJOHN, W. S. NORRIS, Ph.D., R. PITKETHLY, A.I.C., G. SELL, H. G. SHATWELL, Ph.D., R. STANSFIELD, A.M.Inst.C.E., H. E. TESTER, B.Sc., W. H. THOMAS, A.R.S.M.

Chemistry of Petroleum.

274. METHANOL FROM HYDROGEN AND CARBON MONOXIDE. II. DIMETHYL ETHER. R. Brown and A. E. Galloway. *Ind. Eng. Chem.*, 1929, **4**, 310.

The catalytic production of methanol from a mixture containing two parts of hydrogen and one part of carbon monoxide, under a pressure of 180 atmospheres, has been studied over the temperature range 300°-400°C. The catalyst used during the experiments was normal zinc chromate. Employing a space velocity of 3,000, the conversion of carbon monoxide to methanol was found to increase from about 7 per cent. to a maximum of 30 per cent., at a temperature of 375°C., and then to diminish to approximately 20 per cent. at 400°C. Dimethyl ether was found to be an important product of the reaction, being produced from the methanol in quantities proportional to the temperature increase between 300°C. and 400°C. H. E. T.

275. OXIDATION OF PARAFFIN HYDROCARBONS. G. Egloff and R. E. Schaad. *Oil and Gas J.*, 1929, **27**, **43**, 156.

In this paper the authors give an account of the various organic chemicals, which can be obtained from gaseous hydrocarbons, the gaseous hydrocarbons being obtained from natural gas, crude oil, cracking of heavy oils and carburetted water gas. By oxidation of these hydrocarbons the following products may be obtained, methyl alcohol, ethyl alcohol, propyl alcohol, butyl alcohol, formaldehyde, acetaldehyde, propionaldehyde, together with formic, acetic and butyric acids. The oxidiation of hydrocarbons provides a complex problem, although one result of the oxidation of hydrocarbons on a large scale is the formation of a mixture of formaldehyde and methyl alcohol as a by-product in the production of carbon black from natural gas. Researches carried out since earliest times indicate that hydrocarbons undergo diverse oxidation reactions, yielding nearly every possible theoretical product. In the literature on the oxidation of hydrocarbons, we find in addition to the ultimate products of the reaction, namely, water and carbon dioxide, the identification of hydrogen, carbon, carbon monoxide, alcohols, aldehydes, acetone, acids, saturated and unsaturated hydrocarbons, resins and traces of hydrogen peroxide. The difficulty of controlling oxidation reactions may be attributed to the greater ease with which the primary products of the reaction become oxidised compared with the hydrocarbons themselves. A complete summary is given in tabular form of all the compounds which have been identified among the reaction products during the oxidation of methane, ethane, propane, butane and iso-butane.

The paper concludes with a useful summary of the outstanding research work which will have to be carried out before our knowledge of the mechanism of oxidation of hydrocarbons, together with its commercial possibilities, will be complete. H. E. T.

276. THE MECHANISM OF THERMAL DECOMPOSITION OF THE NORMAL PARAFFINS. E. N. Hague and R. V. Wheeler. *J. C. S.*, 1929, pp. 378-391.

An account is recorded of the mechanism of thermal decomposition of the normal paraffins, methane, ethane, propane, n-butane, n-pentane, n-hexane, together with ethylene and acetylene. From the action of heat on the unbranched paraffins from ethane to n-hexane it was concluded that the primary decompositions could be represented by a series of equations, indicating the rupture of the hydrocarbon chain in any position, with the production of an olefin and the complementary lower paraffin or in the limit, hydrogen. Thus with ethane, propane and n-butane the authors have found the primary decompositions to be represented by the following equations:

Ethane: 1. $CH_3 \cdot CH_3 = CH_2 : CH_2 + H_2$.

Propane: 1. $CH_3 \cdot CH_2 \cdot CH_3 = CH_2 : CH_2 + CH_3 \cdot H$.
2. $CH_3 \cdot CH_2 \cdot CH_3 = CH_3 \cdot CH : CH_2 + H_2$.

n-Butane: 1. $CH_3 \cdot CH_2 \cdot CH_2 \cdot CH_3 = CH_2 : CH_2 + CH_2 \cdot CH_2 \cdot H$.
2. $CH_3 \cdot CH_2 \cdot CH_2 \cdot CH_3 = CH_3 \cdot CH : CH_2 + CH_3 \cdot H$.
3. $CH_3 \cdot CH_2 \cdot CH_2 \cdot CH_3 = CH_3 \cdot CH_2 \cdot CH : CH_2 + H_2$.

As the hydrocarbon series is ascended, the tendency for hydrogen to be eliminated rapidly diminishes. Following the primary decomposition, many complicated reactions occur during pyrosynthesis at higher temperatures. Thus, with ethane at temperatures above 700°C., the hydrogenation of fugitive "residues": CH_2 appears to occur (Bone and Coward, *J. Chem. Soc.*, 1908, 93, 1197), while at comparatively low temperatures subsequent to the formation of ethylene, butadiene is produced from each of the paraffins. Above 700°C., under the conditions of the experiments, benzene and other aromatic hydrocarbons are produced from each gas. The experiments show that ethylene readily polymerises to butylene, which by elimination of hydrogen yields butadiene. The synthetic products obtained from the paraffins were found to contain six membered unsaturated hydrocarbons boiling over the range 59 deg. to 78 deg., and hence it was concluded that the butadiene reacts with the ethylene.

Methane, the first member of the paraffin series, was found to have a decomposition point approximately 200°C. higher than that of ethane, while there is only an interval of 25°C. between the decomposition points of the other hydrocarbons. The optimum temperature for the formation of benzene from methane is 1,000°-1,100°C. The main decomposition products of methane are carbon and hydrogen, but at comparatively low temperatures ethylene is produced, which is regarded as the source of the aromatic hydrocarbons.

With each hydrocarbon three types of experiment were made, (1) static; (2) circulation; (3) stream, in which large volumes of gas were passed through a tube of quartz at a constant rate and at a constant temperature.

The most striking feature of the results obtained is the similarity in behaviour of all the paraffins at high temperatures. Below 750°C., when the primary decompositions are taking place, each hydrocarbon follows its own decomposition course, but at higher temperatures the secondary reactions are the same for all. The temperature range 700°-750°C. is the transition

period, and within this range all the normal paraffins, with the exception of methane, give a maximum yield of ethylene. Under similar conditions, the same types of aromatic hydrocarbons are obtained throughout the series from ethane to n-hexane. H. E. T.

277.—The Decomposition of Methane. Part I. C. W. H. Jones. J.C.S. 1929, pp. 419 421.

This research is an investigation on (*a*) the thermal decomposition of methane under various conditions of temperature, pressure, and times of heating and (*b*) the behaviour of the gas when subjected to the action of the electric spark. The methane used was obtained as natural gas from a South Wales colliery, and contained 92·5 per cent of methane, 3·5 per cent. of hydrogen and 3·9 per cent. of nitrogen. Analyses were carried out in a Bone and Wheeler apparatus, following the scheme of Williams-Gardner (*Fuel*, 1925, **4**, 430).

The decomposition of methane when passed through a heated quartz tube is mainly into its elements, although small quantities of olefinic and acetylenic hydrocarbons are also formed. The temperature range investigated was 700°—1080°, the rates of flow of gas being 100, 200 and 300 cms. per second, and the pressures between 10 and 70 cm. of mercury. At comparatively low temperatures, only traces of olefinic hydrocarbons are formed, but at 1,000° and higher temperatures appreciable quantities are produced. Small amounts of acetylene are formed at all temperatures, the maximum yield occuring at a pressure of 30-40 cms. Easily condensable hydrocarbons were not produced under the experimental conditions.

For the experiments on the sparking of methane, the apparatus consisted of an explosion pipette of about 200 c.c. capacity, fitted with platinum wire electrodes and connected to a constant volume manometer. From the results obtained it is concluded that the reaction is mainly bimolecular, tending to become more complex as the reaction proceeds. From the results of analysis of the sparked gas, the main reaction was found to be direct decomposition into carbon and hydrogen, but high percentages of olefinic and acetylenic hydrocarbons are produced. The formation of olefinic and acetylenic hydrocarbons is rapid at first, but ultimately tends to reach a steady level. The yield of olefins appears to be proportional to the gas pressure, but the yield of acetylenic hydrocarbons reaches a maximum value at a pressure of half an atmosphere. Small quantities of complex solid and liquid hydrocarbons were formed on the walls of the explosion pipette. H. E. T.

278.—Studies on Inflammability of Hydrogen. Tanaka and Nagai. Proc. Imp. Acad., Japan. 1928. **4**, 4.

The authors examine the influence of ethyl ether, ethyl alcohol, acetone and various hydrocarbons on the limits of inflammability of hydrogen-air mixtures. The limits were determined in a glass tube 5 cm. diameter and 65 cm. long, both ends of the tube being kept closed and the flame being made to propagate downwards. The tests show that small additions of the materials used lower the upper limit of inflammability of hydrogen-air mixtures very markedly and that greater additions lower the limit more slowly. If the upper limit of hydrogen be assumed to be 60/63 per cent., instead of 71 per cent., Le Chatelier's law holds well in mixtures rich in the vapour of the compounds added.

It is suggested that it is possible to make hydrogen less dangerous for aeronautical purposes by the addition of suitable other combustibles in small proportions. R. S.

279.—Hydrocarbon Emulsions and their Industrial Applications. L. Meunier. Chim. et Ind., 1929, 21 (1). 3-19.

A description is given of the general preparation of emulsions and the influence of the emulsifying agent on the stability of the emulsion.

In industry, emulsions are prepared generally by one of three methods :—(*a*) by "Mixers," (*b*) in colloid mills, and (*c*) by forced circulation. Method (*b*) produces the finest emulsion. High temperature decreases the viscosity of one of the phases but lowers the interfacial tension between the two phases.

Two types of emulsions met with in industry are those of oil in water and water in crude petroleum. Type one comprises hydrocarbon emulsions, emulsions formed by the water of condensation of steam engines, those of bitumen and tar used in road-making, and latex of caoutchouc etc. The emulsion can be eliminated by three methods, viz., mechanically by filtration, chemically by adding an electrolyte whose cation has a high valency, or electro-chemically using iron electrodes. Latex of caoutchouc is an emulsion denoted by the formula $(C_{10}\ H_{14})n$ whose particles are negatively charged. Alkalis stabilise it, but acids tend to coagulate it by neutralising the charge.

The second type of emulsion is formed by infiltration of water during the drilling of the borehole and must be removed before distillation. The asphalt becomes absorbed by the clay material and this mixture constitutes a protective film around the water globules. On the other hand the presence of fatty and naphthenic acids in petroleum decreases to a great extent the interfacial tension—petroleum/water. The emulsion formation is also increased by the sulphonation of naphthenic acids in refining. It can be eliminated mechanically by the application of heat, by chemical means or finally by electrical methods. In the mechnical process a special centrifuge rotating 17,000 times per minute is used. The action of heat (50°-70°C) is suitable for "bottom settlings" occurring at the bottom of reservoirs. Californian crudes are treated at 175°-260°C and under a pressure of 3-3·5 atmospheres. Chemical means consist of adding an electrolyte, while electrical methods neutralise the charge of the water globules, thus causing a rupture of the protective film.

The emulsion of fatty bodies is next discussed. Two phases are present here, viz., fatty acid phase and aqueous phase. The three hydroxyl groups in glycerin remain in the water whilst liberated fatty acids which have a polarity for water less than that of a neutral glyceride, remain in the oil. The surface of separation must be increased to a maximum to bring about hydrolysis of the fatty substance.

In industrial saponification four methods are adopted :—

1. Saponification by steam under pressure, the emulsion is produced by violent agitation.

2. Saponification by alkali—the immediate neutralisation of fatty acids determines the formation of a corresponding quantity of soap available to emulsify the mass of neutral fatty substances, and so causes hydrolysis. The liberated fatty acids increase the proportion of soap contained in the mixture and decrease the quantity of free alkali. The development of the emulsion and consequently hydrolyis is thus accelerated.

3. Hydrolysis brought about by a pre-treatment with concentrated acid gives rise to the formation of sulphonated glycerides which are very good emulsifying agents. On adding boiling water, there is an immediate formation of an emulsion and rapid hydrolysis ensues.

4. Hydrolysis effected by Twitchell's reagent whose exact composition is unknown. It seems to consist of a mixture of sulphonated derivations of fatty acids and aromatic carbides. W. S. C.

280. Production of Low-boiling Hydrocarbons. W. Gessmann and E. W. Shalders. E.P. 299,820, June 30, 1927.

Low-boiling hydrcarbons are produced by passing a mixture of an unsaturated hydrocarbon, preferably acetylene, carbon monoxide and hydrogen, without application of heat and at about normal pressure, over a metal catalyst which is subjected from time to time to the influence of chemically active rays of light. The catalyst consists of pure, powdered electrolytic copper, mixed with tungsten and powdered pumice, the mixture being treated with dilute caustic soda solution and then dried thoroughly. A typical catalyst is composed of 30 per cent. copper, 15 per cent. tungsten, 49 per cent. pumice, 4 per cent. lithium carbonate and 2 per cent. caustic soda.

H. G. S.

281. Production of Butadiene Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenindustrie Aktiengesellschaft. E.P. 307,945, March 11, 1929.

Butadiene hydrocarbons are produced by heat treatment of artificial or naturally occurring open-chain or cyclic paraffins or olefines in the gaseous or vapour state in dilution with at least 50 per cent. by volume of water vapour (not derived from the material under treatment) in the presence of the catalysts specified in E.P. 297,398 or of silica, silicates or noble metals. Those parts of the apparatus which become hot are preferably made of or lined with base metals which do not cause any considerable deposition of carbon. A steel containing about 72 per cent. iron, 7 per cent. nickel and 19·6 per cent. chromium is particularly advantageous.

Thus, 1 vol. of *cyclo*hexane vapour and 0·5 vol., or more, of water vapour are passed at 680° over a catalyst of coarse grained calcium oxide; the yield of butadiene is about 65 per cent. of the theoretical.

Tetrahydrobenzene vapour and an equal weight of water vapour are passed at 650° over magnesium oxide. About 40 per cent. of the tetrahydrobenzene is decomposed, and of this 85 per cent. is converted into the amount of butadiene theoretically possible and a smaller proportion into butylene, propylene and ethylene. In all about 90–95 per cent. of the decomposed tetrahydrobenzene is converted into olefines, the remainder passing to methane and hydrogen.

W. S. N.

282. Manufacture and Production of Liquid Hydrocarbons from Olefines. J. Y. Johnson, Assr. to I. G. Farbenindustrie Aktiengesellschaft. E.P. 309,199. March 28, 1929. Appl., Sept. 28, 1927.

It has already been proposed (E.P. 291,137) to use precious metals or copper in the form of coherent surfaces as catalysts for the production of liquid hydrocarbons from ethylene. It is now found that metals in the coherent state, *e.g.*, iron or copper, superficially coated by heat treatment with tin, zinc, aluminium or chromium give excellent results. The reaction tube itself may be internally coated in this way. An additional advantage is that tinned copper tubes do not become brittle as do copper tubes. The use of a heat treatment is necessary because peeling is liable to occur if electrodeposition is employed. Zinc or tin may be applied in the molten condition; aluminium or chromium as a powder and the whole then heated.

At 800°, 100 gm. of ethylene by passage through an iron tube coated internally with aluminium yield about 37 gm. of a condensate, 70 per cent. of which boils below 200°. The condensate contains about 5 per cent. of tar (containing naphthalene and anthracene) and about 60 per cent. of aromatic

constituents, the remainder being saturated and unsaturated liquid hydrocarbons. The exhaust gas contains about 45 per cent. of methane. There is no deposition of carbon in the tube.

About 8 gm. of a yellow condensate, free from tar and consisting of about 50 per cent. of aromatic constituents, mainly of low boiling-point (the remainder being saturated and unsaturated liquid hydrocarbons), are obtained from 100 gm. of ethylene by a single passage through a tinned copper tube at 750°. The exhaust gas contains only 3 per cent. of methane and 0·6 per cent. of hydrogen, the remainder being ethylene. No carbon is deposited n the tube.

If gases containing 20–40 per cent. of gaseous olefines, obtained, *e.g.*, by heating coal, tar, or mineral oil at about 800° (*cf.* E.P. 269,593), be employed for the production of liquid hydrocarbons, the operation may be conducted at 800° with a reduced gas velocity in the tube without carbon being deposited or tar being formed. W. S. N.

See also Abstracts Nos. 308, 312, 326, 327 and 331.

Crude Oil.

283. Special Processing of Coking Crudes. G. W. Cupit, Jr. *Refiner*, 1929, 8 (3), 71.

This paper, the second of a series, deals with the processing of sour distillates, or the intermediate fraction between water white distillate and gas oil. This distillate has a gravity of 37–38.5° Bé, and an assay distillation shows a boiling range of from 170–178° F. to 710–720° F. The first process is washing the dry oil with sulphuric acid of 93 per cent. strength, with subsequent neutralization and washing. Assay distillation then shows a boiling range of from about 278° F. to 610–620° F. The cut is distilled, an overhead equivalent to 47–49 per cent. of the charge being taken. This overhead should show an initial boiling point of about 130° F., 50 per cent. over at 284° F., and a final boiling point of about 437° F. The oil may be sweetened by treatment with sodium plumbite solution, or it may be blended with blending naphtha and purified by filtration through a No. 1 clay.

The residue of this distillation is re-run for blending naphtha (43 per cent.), 42–38° Bé distillate (4 per cent.), and 40–36° Bé gas oil (Trade gas oil). The blending naphtha has an initial boiling point of 260° F., and a final boiling point of 466° F.

The article then discusses the processing of standard white distillate. It is acid washed using 7 lb of 93 per cent. acid per barrel, and after neutralisation and washing is redistilled from a fractionating tower still. The various types of cuts obtainable are discussed, typical products being :—

	Standard White Distillate	Motor Gas.	Blend Naphtha	Kerosene.	Gas Oil.	Loss.
	100%	19.5%	13.7	57.6%	5.8%	3.4%
Gravity (° Bé) ..	42.5	48.2	44.6	40.8	34.3	—
Initial Boiling Point (° F.) ..	358	306	370	425	575	—
Final Boiling Point (° F.) ..	516	416	421	490	620	—
50% off (° F.)	426	348	378	447	540	—

Paraffin distillate is next considered. It is washed by means of milk water obtained by the neutralization and washing of acid treated paraffin oils,

either red or pale. An activator, preferably foots oil, is added to the distillate, and it is then slowly cooled to 12–14° F. At this temperature it is pressed first for an hour or so at 150 lb pressure, and finally at 300–350 lb for 12 to 20 hours. W. L.

284. The Refining of Muskegon Crude Oil. Faragher and Morrell. *Petr. Times*, 1929, 21, **538**, 807.

Methods of refining Muskegon crude, involving the use of the cracking process, that will permit the profitable handling of this crude are presented in this article. The crude oil produced from the Dundee sand presents a most unusual refining problem and differs from that met in the refining of Lima crude oil. With the latter, the chief problem was that of desulphurisation, and was met by the Frasch copper oxide process. With Muskegon oil, the difficulty is the making of a motor fuel of good anti-knock value, and to a less extent, the making of a marketable product by ordinary refining methods. An analysis of the crude is given, together with the yields of products obtained by straight distillation and by cracking. The straight run gasoline contained only 0.02 per cent. of sulphur, but the material was very refractory to the usual refinery methods for sweetening. Continuous washing with sodium plumbite solution failed to give a sweetened product. Hypochlorite did not sweeten the gasoline, and blowing the material with air in the presence of plumbite solution and lead sulphide sludge failed to convert the mercaptans into disulphides. A satisfactory treatment was developed by blending the straight run gasoline with the cracked pressure distillate in the proportion made from the topped crude and the cracking stock. The blend was treated and re-run as follows:—(1) Wash with about 2 per cent. of 30° Bé plumbite solution, and remove the sludge. (2) Wash with water. (3) Agitate with 3lb. barrel of 66° Bé sulphuric acid and remove sludge. (4) Wash with water. (5) Neutralise with 1 per cent. by vol. of 16° Bé sodium plumbite solution and remove sludge. (6) Distil to specification with fire and steam. From engine tests carried out on the straight run gasoline it was apparent that the obstacle to making a marketable product from Muskegon crude was the knocking properties of the spirit. It is shown that a 350° E.P. gasoline with a benzol equivalent of +10 representing 20.5 per cent. of the crude can be prepared, equal to the average Mid-Continent straight run gasoline. It is established that the cracking process makes possible a complete utilisation of the hitherto unprofitable Muskegon crude. W. H. T.

285. Apparatus for Fractional Distillation of Tar, Crude Oils, Petroleum, and Other Liquids. O. Elstermann and A. Baumhör. E.P. 307,243. April 30, 1928.

Several distillation units are superimposed in a column, each unit being funnel-shaped. The units are heated to various temperatures and are connected by passages from the lower pointed end of each funnel to the funnel immediately below. Rotary distributors are provided, and these throw the liquid on to the inner walls in thin layers so as to flow in a helical path while being fractionated. G. S.

286. Reclaiming Crude Oil. H. V. Meathers. U.S.P. 1,705,370. March 12, 1929.

An improved process and apparatus are described, for reclaiming crude oil from basic sediment oil, similar to that usually found at the bottom of reservoirs used for storing crude oil in the oil fields. With the apparatus described, it has been found that a very high percentage of crude oil can be recovered from comparatively old basic sediment oil. H. E. T.

See also Abstracts Nos. 279 and 354.

Natural Gas.

287. H_2S Only need be removed from Gas. D. L. Jacobson. *Oil and Gas J.*, 1929, **27** (46), 116.

The article commences with a brief survey of the hydrogen sulphide and organic sulphur content of a number of crude oils and sour natural gases. A table is included showing the results of the analyses of several refining gases for the same components. Available data indicates that the quantity of organic sulphur compounds in natural gas or refinery gases rarely, if ever, exceeds 8 grains per 100 cub. ft., an amount that is well below the legal limit of 35 grains per 100 cub. ft.

The corrosive action and general deleterious effects of hydrogen sulphide are discussed, first when the gas is to be used as fuel (domestic or industrial) and then when the gas is to be used for gasoline recovery.

A brief description of the Seaboard method of liquid purification of gas closes the article. In this process the gas is washed with a 1 to 3 per cent. solution of sodium carbonate, and it is claimed that about 90 per cent. of the hydrogen sulphide is removed in one absorber, and that if two absorbers are used in series 98 to 99 per cent. of the hydrogen sulphide is removed. The spent alkaline liquor is reactivated by air blowing. W. L.

288. Distillation Apparatus in Use in natural Gasoline Manufacture. J. E. Kobednick. *Oil and Gas J.*, 1929, **36**, 132.

The progress made in the design and construction, of a distillation unit for the production of natural gasoline is outlined. Laboratory tests and plant practice have proved that the absorption oil used in the manufacture of natural gasoline should be comparatively light without containing the same constituents as the gas. Such oils are better absorption media than the heavier oils commonly used, and besides their absorption qualities they have lower pressure drops through the heat interchangers, cooling coils and piping.

The vertical steam still is best suited for economical construction and operation. The basic principles for which the still should be designed are :

1. To strip the oil of its gasoline fractions.
2. To fractionate the vapours, so that only gasoline fractions are delivered to the final gasoline condensers.

In a well designed column both of these phases may be accomplished, although the work of each phase is different. A column capable of stripping the oil and also separating the heavier constituents from the vapours, as well as permitting "end-point control," would consist of a stripper section and a fractionating section. The feed to the stripper is preheated, in order to effect spontaneous vaporisation of most of the lighter fractions, in the compartment where the feed is introduced, while the heavier remaining fractions are differentially stripped in the stripper. The size of the stripping and fractionating sections must be designed for an adequate liquid and vapour capacity. H. E. T.

289. Heat Treatment of Hydrocarbon Gases. Anglo-Persian Oil Company, Ltd., A. E. Dunstan and R. V. Wheeler. E.P. 309,455 Apr. 8, 1929. Appl., Oct. 8, 1927.

For the production of aromatic hydrocarbons, a stream of gas containing gaseous paraffins is gradually heated to a temperature (*e.g.*, below 550°) substantially below that at which eventual decomposition is to be carried out. It is then heated in a determined course to a temperature (650—975°), at which the paraffins are decomposed and aromatic hydrocarbons are

formed. The stream of gas is then suddenly reduced in velocity, *i.e.*, expanded, and cooled and the free carbon formed is deposited; finally, the aromatic hydrocarbons are condensed. If necessary, the original gas is previously freed from hydrogen sulphide in known manner, *e.g.*, by limited oxidation with air or sulphur dioxide at 250—300°, and from condensible hydrocarbon vapours. The yield of aromatic hydrocarbons is increased by diluting the gas with an inert gaseous material (preferably steam, which may advantageously be superheated), which is preferably introduced after the gas has been pre-heated. Thus, the addition of about 25 per cent. by vol. of steam gives an increase in the liquid yield of about 100 per cent., and a decrease in the carbon formed of about 30—50 per cent. The reaction temperature is controlled according to which paraffin or mixture of paraffin is present. The temperatures preferred are 850—975, 800—900, 750—850, 700—800 or 650—750° for methane, ethane, propane, butane or pentane, respectively; for mixtures, a range of temperature is chosen according to the proportions of the constituents. The velocity of the gas in the preheater and reaction tubes is so maintained that the flow is turbulent, giving good heat transference and maintaining the carbon particles in suspension. Thus, 1,000 c. ft. of gas, *d* 1·69 (air 1·0), gave on thermal decomposition at 800° in the presence of about 25 per cent. by vol. of steam, 1·75 gal. of light spirit, *d* 0·875, final b.p. 140°, 0·64 gal. of tar, *d* 1·09, about 2,500 c. ft. of gas, *d* 0·5, and about 9 lb. of carbon. The spirit had 80 per cent. of b.p. 79·5—85°: the tar contained 15—30 per cent. of aromatic liquids b.p. below 150°. W. S. N.

290.—Apparatus for Preparing Compressed Gases. Gesellschaft für Industriegasverwertung mit beschränkter Haftung. E.P. 279,041, Sept. 3, 1927. Conv. (Germany), Oct. 18, 1926.

Gases of different controllable pressures are prepared from the liquid state in a pressure vessel provided with a thin-walled inner vessel for the reception of the liquefied gas and surrounded by a tubing coil which dips into the liquid. The distance of the tubing from the exterior of the pressure vessel increases progressively in the direction of liquid flow. G. S.

291.—Synthesis of Liquid Hydrocarbons. H. E. Potts. E.P. 309,002. April 2, 1929.

This invention relates to a process for the synthesis of liquid hydrocarbons obtained by subjecting gaseous carbon compounds to the action of an intense beam of β-rays of velocity exceeding one-half that of light. The gases used are preferably water gas, coke oven gas, natural gas or gases produced during the cracking of oils. The β rays, while in the presence of the reacting gases, are subjected to the action of an intense electric or rotational magnetic field. The activated gases, during or after irradiation, are preferably subjected to the action of high pressures or temperatures: the latter producing a ripening or after reaction. Catalysts may be used in order to accelerate the reactions. Suitable catalysts are those known in the heat-pressure synthesis of liquid hydrocarbons from water-gas. It is possible to increase the yield of liquid hydrocarbons obtained by circulating the reacting substances under increased pressure. H. E. T.

292.—Recovering Volatile Oils from Gases containing the Same. S. Yamamoto. U.S.P. 1,710,284 and 1,710,285, April 23, 1929. Appl. May 18, 1926, and June 22, 1926. Japan, March 3, 1926.

Two absorption towers, arranged to work alternatively, are connected in a closed cycle to a cooler, a fan, a heater, a by-pass to cut off the heater and a

boiler to supply the heating medium. Each tower in turn is utilised in the absorbing operation while the other, already saturated with oil vapour, is subjected to the recovery operation in which absorbed oil vapour is removed from the absorbent contained in the tower by a stream of hot gas from the heater. This hot gas, rich in oil vapour, is cooled in the cooler, any condensate collecting in a receiver. The permanent gas, drawn by the fan from the cooler, passes through the heater and again acts to drive off the oil vapour still remaining in the tower. When this is complete, the heater is cut out of the cycle and the tower cooled by cold gas. The tower is then transferred to the absorbing system and the alternative tower brought into the recovery cycle. H. S. G.

293.—Combining Natural Gas and Hydrocarbon Oil for the Production of gasoline. L. S. Worthington, Assr. to C. L. Thompson. U.S.P. 1,705,348. March 12, 1929.

The invention contemplates the spraying of a jet of atomised oil into a current of gas, the oil and gas both being heat treated prior to mixing, in order to produce a proper working temperature. The vapours are condensed to produce a fuel the burning of which is practically without carbon deposit. Another feature of the process includes the use of a catalyst such as uranium, vanadium, thallium or molybdenum, or the introduction of air at a desired temperature. Additional agents may be included into the atomised mixture, and these may include those already mentioned together with gases of combustion (carbon dioxide, carbon monoxide, etc.). A sketch of the apparatus suitable for carrying out the process is given. W. H. T.

See also Abstracts Nos. 275, 337, 338, 339, 342, 356 and 417.

Motor Spirit.

294. Relation of Fuel to Engine Acceleration. D. B. Brooks. *A. P. I. Bull.* 1929. 10–2. 143.

The report of 1928, *A. P. I. Bulletin*, Jan. 31, p. 74, referred to work commenced to determine the significance of the portion of the A.S.T.M. distillation curve between the 20 per cent. point and the 90 per cent. point. Later tests showed that the methods of measurement of acceleration needed to be more precise. A new instrument has been devised in which a paper tape moved by a sprocket driven from the engine is punctured by a spark controlled by a tuning fork. Details of the apparatus are given in an appendix.

The acceleration tests were made on a 6-cylinder 3-port overhead valve engine, 3½ in. bore by 4½ in. stroke, coupled to a Sprague electric dynamometer arranged to give conditions of level road operation in high gear.

In order to make it possible to interpret the results, the carburetter was modified to eliminate the accelerating well and to include means for controlling the air-fuel ratio. Ignition advance was adjusted to suit the maximum power mixture at the mean speed of the acceleration range and was retained constant for all tests reported.

In order to correlate the speed-time curves used for the previous report with the acceleration-speed curves now used a typical comparison is given.

The general effect of air-fuel ratio on acceleration is that richer mixtures reduce the initial lag, but beyond a certain point the performance at higher speeds is also reduced. An air fuel ration of about 13 to 1 with a suitable accelerating charge gives the maximum performance throughout.

The principal effect of increasing the temperature of the manifold is also to reduce the initial lag, and acceleration at lower speeds is much improved. The performance at higher speeds falls away slightly.

Increase of jacket temperature reduces the acceleration considerably, probably due to decreased volumetric efficiency. A much better initial acceleration is obtained with aviation gasoline than with commercial gasoline at low speeds, but the difference disappears at about 650 r.p.m. These tests were made with a manifold temperature of 33°C. At a temperature of 92.5°C. the difference is considerably reduced. A blend of 50 per cent. aviation gasoline with 50 per cent. commercial does not show an improvement equal to 50 per cent. of the difference between the two unblended spirits.

Further investigations made with three special fuels which distilled equally at the 20 per cent. and 90 per cent. points, but which differed between these points, show that at low manifold temperatures (—9 to +10°C.) the acceleration is best for the fuel having the highest distillation temperature for the 50 per cent. point. At temperatures of 32° and 42°C. the order is reversed, the acceleration increasing as the distillation temperature for the 50 per cent. point falls. At 98°C. the order remains the same but with a general approach to a normal aviation gasoline.

It is suggested that there may be a relation between the A.S.T.M. curves and acceleration, but that no reliance must be placed on the general applicability of the data given until further work justifies such reliance. R. S.

295. Crankcase Dilution Unimportant Factor in Gasoline Performance. J. M. Evans. *Oil & Gas J.* 1929, **36**, 154.

In this paper the author discusses the effects of crankcase dilution with reference to gasoline performance and efficient lubrication. The trend of research points to the fact that crankcase dilution within reasonable limits is not injurious to an automobile engine. The gasoline fractions which dilute the lubricating oil merely serve to lower the latter's viscosity, and unless this dilution becomes excessive the viscosity will never be lowered to a point at which the bearings are not receiving sufficient lubrication. There are many factors which affect the amount of crankcase dilution found in any particular automobile, among which may be mentioned the quality of the gasoline, the atmospheric temperatures encountered, the type of driving, the condition of the engine and the load. Runs of 500 miles or more were made on a car using various commercial gasolines, and it was found that the dilution gradually increased from zero when a new oil was in the crankcase, to a maximum value after about 500 to 600 miles had been run. The maximum dilution encountered was of the order of 10 per cent., and even when using fuels much heavier than commercial gasolines, such high dilutions as 30 to 40 per cent. were never encountered.

That dilution does reach a final equilibrium point was shown by tests on a particular car as follows: Oil to which 12 per cent. of dilution had been added was placed in the car, and a run was made at 40 m.p.h., samples being taken every 50 miles. The dilution was found to decrease steadily down to a constant value of 4 per cent., indicating that the diluent added was distilling through the breather pipe faster than further dilution came past the pistons. The same car was now run with an oil containing 3 per cent. dilution, when it was observed that the dilution, after running about 20 miles, increased to 4 per cent. and then remained steady. The data thus obtained indicate that the normal car will have a crankcase dilution of approximately 10 per cent., which may ordinarily be expected with any well refined gasoline. H. E. T.

296. Reagent for the Removal of Sulphur. Axtell Research Laboratories, Inc. E.P. 282,738. Mar. 11, 1929.

Sulphuric acid, preferably fuming acid containing a considerable percentage of sulphuric anhydride, is treated with an excess of an aromatic hydrocarbon. The lower acid layer containing sulphuric acid and products derived therefrom by interaction with the hydrocarbon, after separation from the excess of hydrocarbon, is used in place of sulphuric acid in treating hydrocarbon distillates, particularly cracked distillates. The reagent may be employed without further alteration or it may be blended before use with ordinary or with fuming sulphuric acid. Acid saturated with an aromatic hydrocarbon or with a mixture of aromatic hydrocarbons effects the removal or partial removal of sulphur compounds without an undesirable oxidising or polymerising action on the valuable unsaturated hydrocarbons of the oil. The portion of the sulphur compounds not removed is likely to have undergone transformation into comparatively complex sulphur compounds of high molecular weight which, upon redistillation, tend to remain in the residue. The treatment with the saturated acid is combined with other treatments.

Thus, a cracked distillate of final b.p. 437° F. and containing 0·68 per cent. of sulphur is given a 25 lb./bbl. treatment with a reagent made by saturating fuming sulphuric acid with "benzene" at normal temperatures. After separating the acid sludge the distillate is agitated with 4 lb./bbl of Death Valley clay, and is then filtered. The practically colourless filtrate is agitated with about 3 lb./bbl. of 33 per cent. aqueous soda solution. The treated oil (85 vols.), containing 0·30 per cent. of sulphur, is mixed with 15 vols. of kerosine distillate of b.p. range 375–550° F. and containing 0·08 per cent. of sulphur, and the blend distilled in the presence of a small quantity of aqueous potassium hydroxide until a vapour temperature of 225° F. is reached. Steam is then introduced and distillation continued until 85 vols. of oil distillate are obtained. The product is satisfactory in colour, odour, etc., and contains 0·11 per cent. of sulphur.

A cracked end-point gasoline, containing 22 per cent. of unsaturated hydrocarbons and 0·79 per cent. of sulphur, some of which is present as sulphur compounds extremely resistant to the ordinary sulphuric acid treatment, is agitated with 25 lb./bbl. of a reagent prepared by saturating fuming acid (20 per cent. SO_3) with commercial benzene and diluting the saturated acid with an equal weight of the original fuming acid. The acid-treated oil (loss 2·9 per cent.) is then treated as in the previous example. Before the final distillation the sulphur content is 0·26 per cent.; after distillation it is 0·09 per cent. and the content of unsaturated hydrocarbons is 21·2 per cent.

For refining a straight-run kerosine distillate, 5–15 lb./bbl. or even more of the saturated acid reagent is employed, or of a mixture of the same with more or less fresh sulphuric acid. W. S. N.

297. Removing Sulphur Compounds from Petroleum Oils. W. N. Davis, W. H. Hampton and E. N. Klemgard, Assrs. to Standard Oil Co. of California. U.S.P. 1,705,809. Mar. 19, 1929.

Cracked naphtha or gasoline is treated to remove sulphur compounds with minimised polymerisation of non-sulphurous constituents by cooling to below 38° F. and bringing into contact with fuming sulphuric acid of m.p. below the temperature of reaction; a continuous, counter-current system may be adopted. W. S. N.

298. Refining Mineral Oils. A. Lachman. U.S.P. 1,709,315. April 16, 1929.

A method is described for purifying mineral oils obtained by distilling or cracking, such as lubricating oils, kerosine, gasoline and shale oils. The invention consists in treating the oil to be refined with phosphoric acid, and in some cases with subsequent small quantities of sulphuric acid and sodium hypochlorite. Gasoline treated in this manner will be stable as to colour on exposure to air, will be water white, and furthermore will require smaller quantities of acid, and will reduce crude gasoline losses to a maximum value of 2 per cent. The gasoline is passed in the vapour state through a mass of phosphoric acid, when nearly all the impurities are removed. The refining operation is then completed by condensing the hydrocarbon vapours and treating the condensate with a strong alkaline solution of sodium hypochlorite. During the refining operation the strength of the phosphoric acid is maintained at approximately 85 per cent. and its temperature at 200° C. Diagrams of the apparatus employed in carrying out this invention are appended. H. E. T.

See also Abstracts Nos. 280, 283, 293, 336, 337, 338, 339, 340, 341, 342, 357, 358, 359, 360, 361, 362 and 383.

Kerosine.

299. Process of Treating Hydrocarbons. J. C. Black and M. L. Chappell. U.S.P. 1,710,143. Apr. 23, 1929. Appl. Apr. 6, 1927.

This invention relates more specifically to the treatment of lamp-oil distillates from a petroleum oil of the carbocyclic or asphalt base series. It consists in the purification of these distillates, first, by extracting a part of the undesired hydrocarbons with liquid sulphur dioxide at low temperatures (about 17°F.), and then immediately finishing the purification, at the same low temperature and in the presence of dissolved liquid sulphur dioxide, by the employment of sulphur trioxide or fuming sulphuric acid.

It is claimed that the process gives a substantial reduction in refining losses. W. L.

300. Process for Treating Hydrocarbon Oils. J. C. Black and M. L. Chappell. U.S.P. 1,710,200. Apr. 23, 1929. Appl. Mar. 10, 1927.

This invention relates to the treatment of petroleum oils or distillates, including those from thermo cracking processes, with sulphuric acid in the presence of sulphur dioxide. The latter is dissolved in the oil at relatively low temperatures (approximately 17°F.) and under sufficient pressure to retain it in the oil in a liquid state. The amount of sulphur dioxide used is approximately 25 per cent. by volume on the oil.

The washing of the oil, containing the requisite amount of sulphur dioxide, with sulphuric acid may be done intermittently or continuously by any preferred method.

It is claimed that by this process the loss of stable unsaturated hydrocarbons is minimised as in the presence of sulphur dioxide and at relatively low temperatures, the chemical action of the sulphuric acid is modified and a selective action obtained, the acid reacting substantially only with the unstable unsaturated and sulphur bearing hydrocarbons with the formation of sulpho-acids or sludge. W. L.

301. Process for Purifying Hydrocarbons. J. C. Black and M. L. Chappell. U.S.P. 1,710,201. Apr. 23, 1929. Appl. May 28, 1927.

Petroleum oil distillate, derived from an asphaltic or mixed base crude oil, is first treated, by counter current flow, with liquid sulphur dioxide in

quantities sufficient to extract a major portion of the undesired hydrocarbons without substantial extraction of the desired ones, the temperature being maintained at about 17°F.

The partly purified oil containing dissolved liquid sulphur dioxide is separated from the surplus liquid sulphur dioxide and treated with sulphur acid or fuming sulphuric acid in quantities sufficient to complete its purification. This acid wash is performed at temperatures below 60°F.

A selective treatment is obtained, the low temperature treatment with sulphuric acid in the presence of a reducing agent preventing oxidation and excessive suphonation.

The process is applicable to lamp, transformer, lubricating, medicinal, spray oils, etc., and it is claimed that refining losses are decreased by about 8 per cent. of the volume of the treated oil. W. L.

302. Treatment of Hydrocarbons with Liquid Sulphur Dioxide. E.P. 281,337. Nov. 28, 1927. Conv. (Germany), Nov. 26, 1926.

This invention is an improvement in the known process of treating hydrocarbons with liquefied sulphurous acid in that the requisite low temperatures and pressures necessary for the cooling and evaporation of the used sulphur dioxide are obtained by the use of jet pumps instead of the usual somewhat costly compressors.

The plant is so designed that the gaseous SO_2 obtained from the first stage evaporators by the application of heat to the refined oil and the SO_2 extract operates jet pumps of the ejector type, which produce the requisite low pressure or vacuum for the second and third stages of the evaporation and also for the cooling of the liquid SO_2 in the SO_2 cooler. R. W. L. C.

See Also Abstracts Nos. 283 and 344.

Fuel Oil.

303. Furnace and Fuel Oils Standards. *Petr. Times*, 1929, **527**, 276.

These standards are the recommendations submitted to the industry as a result of a conference held under the auspices of the United States Bureau of Standards. They provide for three grades of oil suitable for use in domestic burners and for three grades for industrial use. If accepted by 65 per cent. of the companies concerned, they will become official United States standards for domestic and industrial fuels.

Furnace or Domestic Oils.

Grade of Oil.	Flash Pt. Min.	Water & Sediment Max.	Pour Pt. Max.	Distillation. Max.	Test.	Viscosity. Max.
No. 1. Light domestic fuel oil. A light distillate oil for use in burners requiring a high grade fuel.	110° F. or legal.	165° F.	0·05%	15° F.	10% Pt. Max. 420° F.	End Pt. Max. 600° F.

FURNACE OR DOMESTIC OILS (*continued*).

Grade of Oil.	Flash Pt. Min.	Water & Sediment Max.	Pour Pt. Max.	Distillation. Max.	Distillation. Test.	Viscosity. Max.
No. 2. Medium domestic fuel oil. A medium distillate oil for use in burners requiring a high grade fuel.	125° F. or legal.	190° F.	0·05%	15° F.	10% Pt. Max. 440° F.	90% Pt. Max. 620° F.
No. 3. Heavy domestic fuel oil. A distillate fuel for use in burners where a medium low viscosity fuel is required.	150° F. or legal.	200° F.	0·1%	15° F.	10% Pt. Max. 460° F.	90% Pt. Max. 675° F. Saybolt Universal at 100° F. 55 secs.

Lower or higher pour points may be specified wherever required by conditions of storage or use, but under no conditions may they require a pour point lower than 0° F.

FUEL OR INDUSTRIAL OILS.

Grade of Oil.	Flash Pt. Min.	Water & Sediment. Max.	Pour Pt. Max.	Viscosity. Max.
No. 4. Light industrial fuel oil. An oil known to the trade as a light fuel oil for use in burners where a low viscosity industrial fuel oil is required.	150° F.	1·0%		Saybolt Universal at 100° F. 125 secs.
No. 5. Medium industrial fuel oil. Same as Federal specification and Board's specification for bunker oil "B" for burners adapted to the use of industrial fuel oil of medium viscosity.	150° F.	1·0%		Saybolt. furol at 122° F. 100 secs.
No. 6. Heavy industrial fuel oil. Same as Federal specification and Board's specification for bunker oil "C" for burners adapted to oil of high viscosity.	150° F.	Water 1·75	Sediment, 0·25%	Saybolt furol at 122° F. 300 secs.

Whenever required, a maximum flash point may be specified. It shall not, however, be lower than 250° F. A maximum pour point may be specified. It shall not, however, be higher than 160° F. W. L.

304. TREATING RESIDUAL OILS. J. C. Morrell. U.S.P. 1,705,199. March 12, 1929.

This invention relates to improvements in the method of treating residual oils, with special reference to the removal of pitch-like, coke forming particles, suspended or partially dissolved in cracked residuum. The residuum while hot is treated with a coalescing agent, such as sulphuric acid, and then agitated, when the coke forming particles coalesce with each other combined with the sulphuric acid and are precipitated from the oil. The sulphuric acid used has a strength of from 90-100 per cent., and reacts either chemically or chemico-physically, the latter causing coalescence of the suspended matter in the oil. The precipitated material containing sulphuric acid derivatives is useful as an asphaltic material. In addition acid sludge from other refining processes may be used as the precipitating material. Full details of the apparatus and method of carrying out the process are given. H. E. T.

See also Abstract No. 346.

Lubricating Oil and Wax.

305. TEST DECOLORISING VALUE OF CLAYS. C. F. Barb. *Oil and Gas J.*, 1929, **27**, (42,) 116, 161, 163.

Twenty-one clays were selected from many sources and a variety of uses, and because many of their properties were well known from industrial usage. The name and origin of the clays are given in Table I., along with the colour of a filtrate when a mineral oil is treated with the clay in the ratio of 3 lbs. per gallon. These results show that three of the clays give very high colours (Tag-Robinson scale) and are very good commercial filters.

Properties of the clays were then examined. The Pinene rise in degrees Centigrade was determined by placing 2 grams of dehydrated clay of 150 mesh in a double-wall test tube, inserting a thermometer reading to tenths of a degree and then pouring in quickly 5 c.c. of pinene. The clay is stirred until thoroughly wet with pinene and the maximum temperature difference is noted. Although not an absolute test, it is probable that a clay which shows a very high temperature rise will be a good filtering agent.

The percentage of malachite green absorbed by a clay is often used as a test of the decolorising power on mineral oils, but the method was not found satisfactory in this work. The most serious disadvantage was that clays containing appreciable amounts of alkali would remove the colour from enormous quantities of dye solution. Further that this colour change is rather an indicator reaction and no precipitates or colloidal particles were apparent. Another troublesome property of the solutions is the rapid fading of the standard solutions made up for comparison, which change may amount to 50 per cent. in two hours.

The results of another dye absorption method is given under the oil green column. The method used is similar to the malachite green method except that the dye is dissolved in a water-white naphtha. The clay must be perfectly dry before it is added to the dye solution. The colour of the solution after agitation is compared with standards made up as in the malachite green method. The solutions do not fade but have a habit of changing colour entirely when used on some clays. This is probably caused by the fact that the dye is not homogeneous but is made up of three constituents which are not absorbed in equal ratios by all clays.

The amount of NaOH absorbed by a clay in aqueous solution seems to give an indication of decolorising value on mineral oils. A 2 gram sample of the powdered clay is shaken up in about 10 c.c. of water and then titrated to a permanent pink colour with N/100 NaOH using one drop of phenolphthalein as an indicator.

The pH values of the solutions formed when the clays are agitated in distilled water and in 5 per cent. = NaCl. solution, along with the water of plasticity and the percentage of shrinkage on drying are also given. None of these properties seem to have any apparent value as tests of a decolorising clay on earth.

In Table IV. is given the chemical composition of those clays for which this information is available along with that of two English earths. The chief difference in the composition of the filtering and non-filtering clays seems to be in the percentage of alumina present. If the amount of alumina in the clay is above 15 per cent., the clay seems to lose its filtering efficiency. The Nutfield, England, earths are of particular value in treating animal fats rather than mineral oils, and it will be noted that the amount of CaO is high. Clay No. 2 had very little effect on mineral oils when in the natural state, but when acid treated and washed it showed the properties similar to No. 1. It appears therefore that if chemical composition has any bearing on filtering efficiencies, the alumina should be low, the alkaline oxides comparatively high. The silicates do not seem to affect the action one way or another. Against this statement is the action of acid-treated clays, which may have the alkaline salts almost completely removed and still be more efficient agents on mineral oils than before.

The proximate analysis of some of these clays has shown something rather more definite than the ultimate chemical analysis. A high kaolin content indicates a low decolorising efficiency of the clay, and this has been borne out in testing kaolins and ceramic clays. In no case has a kaolin shown any value as a filtrant.

Many clays have been tested in both the natural state and again after acid treatment and it has been found that, in general, a clay that shows any value at all as a decoloriser, in the natural state, is but little, if at all, improved by treatment with mineral acids. On the other hand, a clay that is worthless as mined may make an excellent decolorising agent after being digested with acid and washed. The washing with water has been found to be necessary in all cases, whether the clay is to be used as a pulp or in the dry state. It appears that the acid brings into solution certain compounds that saturate the adsorbing material of the clay and these compounds must then be removed by washing. S. W. C.

306. Interfacial Tension Measurements in the Examination of Insulating Oils. J. C. Edwards. *Jour. Scientific Instruments*, 1929, **6** (3), 90–95.

In this paper, a description is given of a method for the determination of the interfacial tension at an oil-acid interface. The device is semi-automatic in operation, and is well suited for routine testing for the examination and comparison of various insulating oils.

The method consists in forming drops of dilute sulphuric acid in the oil at the end of a capillary tube, the size of the drops being determined by counting the number of drops which fall from the end of the capillary, when a known volume of acid is expelled. A number of experiments were carried out to determine whether the apparatus would give reliable and repeatable results. Three transformer oils were used in these experiments, and their interfacial tensions with dilute acid calculated from Tate's law.

K

$$\text{Interfacial Tension} = mg/2\pi r.$$

where r = radius of the tip of the capillary tube in cm.
m = mass of "ideal drop."
= [volume of "ideal drop" in cc.]
× [(Density of acid) − (Density of oil) in grm. per cc.]

From these results obtained it was found that the method gives reliable results and is of use for giving information regarding the state of deterioration, and the probable performance of the oils when in use. H. E. T.

307. Insulating Oils. G. Stadnikoff and Z. Wosschinskaja. *Petroleum*, 1929, **25**, 651–658.

An acid-refined transformer oil may contain sulphonic acids which are extremely difficult to remove by washing, this being also true of their salts. The presence of sulphonic acids or salts thereof may account for discrepancies in the results of previous workers.

The action of oxygen on a transformer oil during the oxidation test gives rise to acids, alcohols and carbonyl compounds. In the presence of a sulphonic acid and of resinous material or gum, *e.g.*, in an insufficiently refined oil, the products of oxidation condense with the resin, this reaction being accelerated by a sulphonic acid or its salts. The condensation products remain in suspension, the excess of resin acting as a protective colloid. The acid number of the oil will remain somewhat low and the oil, according to the oxidation-acid number test, will appear satisfactory. If the oil is used for some time in a transformer, sludge will be formed and the acid number will rise. If, however, the oil contains a sulphonic acid but has been freed from resin by energetic refining, the products of oxidation will be condensed under the action of the sulphonic acid, and the products of condensation, in the absence of gum, will be precipitated, thus a well-refined oil may appear less stable according to the oxidation test than one that contains an appreciable quantity of resin.

These conclusions are based on experiments in which fatty acids are heated in gas oil solution, with and without the addition of a sulphonic acid, in the presence of alcohols, aldehydes or ketones. Besides synthetic acids, volatile acids obtained by the oxidation of a transformer oil at 160° are employed. The presence of a salt of a sulphonic acid in sludge from a transformer oil is demonstrated. W. S. N.

308. Some Characteristics of Amorphous Wax. L. D. Jones and F. E. Blachly. *Ind. and Eng. Chem.*, 1929, **21** (4), 318–320.

In general, the wax which precipitates from the lighter lubricating distillate of a crude oil is crystalline and hard, and the wax which precipitates from the very heavy distillates and cylinder stock residues when they are diluted and chilled is amorphous and soft. Since the character of the wax determines whether a stock can be dewaxed, and by which method, it is important to determine upon what the character of the wax depends. Zaloziecki proposed the theory that petroleum contains soft amorphous wax which is broken down on distillation to hard crystalline wax. Since then, however, Buchler and Graves have shown that wax may be distilled with no evidence of decomposition. The authors separated a soft amorphous wax from a long residuum, and then removed 40 per cent. of this wax by steam distillation and found that the distillate had a pronounced crystalline appearance. When the distillate was reblended with the residue the character of the mixture was not appreciably different from that of the original wax. It has been shown that waxes of low or high melting point, molecular weight, etc., if sufficiently

pure, crystallise in plates. Further, that slack wax that is hard and crystalline deposits plates at the beginning of crystallisation but in the presence of the impurities naturally occurring in slack wax, the edges of the plates roll up, forming needles. It would seem that crystalline form cannot be used as a criterion of the causes which determine the difference between crystalline and amorphous wax.

An oil or an oil solution in which crystalline wax has been precipitated will usually have a frosty surface. In contrast with this an oil solution in which soft amorphous wax has been precipitated shows a clean liquid surface with typical light reflection. This appearance persists even if the mixture be stirred to distribute any amorphous wax that may have settled throughout the liquid.

If a glass rod is dipped into a chilled solution of oil containing a precipitate of soft amorphous wax and then withdrawn, the solution of oil will drain off, leaving only a very thin film of oil adhering to the glass. If the same test is applied to a solution of oil containing a precipitate of crystalline wax, the oil will drain away from the glass rod, leaving crystals of wax adhering to the glass.

An oil solution from which the wax precipitates in an amorphous form may be chilled to —50° F. and with such an amount of wax present that the mixture becomes quite viscous without giving any evidence of coating the chilling surfaces with wax. On the other hand, any precipitation of hard, crystalline wax produced by cooling will result in an immediate coating of wax on the chilling surfaces.

All the characteristic differences between amorphous and crystalline wax which have been described above are dependent upon surface tension. Hard crystalline wax in the presence of an oil solution has appreciable surface tension, but this surface tension is appreciably less than that of the oil solution in which it is suspended, whereas the surface tension of amorphous wax is appreciably greater than that of the oil solution in which it is suspended. It is desirable to account for this difference in surface tension.

Soft amorphous wax normally occurs in petroleum and various residues from the distillation of petroleum whereas crystalline wax occurs in the lighter lubricating fractions. Heavier lubricating distillates contain a semi-amorphous wax, but any method of distillation which tends to improve the degree of fractionation increases the amount of distillate containing hard, crystalline wax. This is notably true of vacuum distillation with bubble towers.

It would seem that the constituent of petroleum which is reponsible for the amorphous character of the wax has a very high boiling point, and by fractionation can be concentrated in a very small fraction of the petroleum. This is still further demonstrated by the action of some heavy lubrication distillate. A lubricating distillate (75–89 per cent. on crude) from a Persian crude oil was found to contain a semi-amorphous wax having marked crystalline characteristics. Another lubricating fraction from the same crude (75–92 per cent.) was found to contain wax as amorphous as the average 600 fire-test cylinder stock. It therefore follows that the substance reponsible for the amorphous character of the wax has a very high boiling point, but that it may be readily distilled without substantial decomposition. Although this substance cannot be shown to be identical with asphalt or colouring substances in the oil, since very light cylinder stocks can be produced which contain quite amorphous wax, nevertheless the substance tends to act in many ways similar to the colouring matter, inasmuch as any reduction of colour results in a reduced softness of the wax when working with distillates containing a semi-amorphous wax.

It is the theory of the writers that in the presence of Fuller's earth or similar agents this substance is adsorbed on the surface of the Fuller's earth, but in the presence of a wax precipitate that it is adsorbed on the surface of the wax crystals, thereby increasing their surface tension and increasing the amorphousness of the precipitated wax.

Some cylinder stocks contain a comparatively hard variety of wax and if the wax is precipitated in fractions by diluting with naphtha, heating to dissolve the wax and chilling 10° C., separating the wax and then chilling 10° C. more and separating the wax, and repeating, the first wax separated will be the hardest and each successive fraction will be softer and more amorphous. This is also true of lubricating oil distillates which contain a wax in the semi-amorphous state. It is suggested that the substance causing the amorphous characteristics of the wax is in true solution or a high degree of dispersion in the naphtha oil solution at higher temperatures, and is not readily adsorbed by the wax crystals at higher temperatures. But at lower temperatures this substance changes to a colloidal state or a lower degree of dispersion, so that it may be more readily adsorbed by the wax, with the result that the wax precipitated at lower temperatures is more amorphous.

It may also be pointed out that some solvents, such as ethylene dichloride, which have a greater solvent action for asphalt and the colouring matter in oils than naphtha, and which therefore probably have a greater solvent action for the substance producing amorphousness, precipitate a much harder wax than is obtained from naphtha solutions. S. W. C.

309. Lubricating Greases (Manufacture by Pressure Method). E. N. Klemgard. *Refiner*, 1929, **8** (3), 68.

The advantages of the pressure method (operated at 50–70 lbs. per square nch) over the old open kettle process consist in greater uniformity of product and much more rapid operation. In grease plants already established, one five- or six-barrel capacity pressure soap kettle can produce sufficient soap base for several 20- or 30-barrel open type grease mixers.

A sectional diagram of a plant consisting of a pressure soap kettle and 1000-gallon open type mixer is given together with the formula and method of operation for the production of a medium cup grease.

A study has been made of the effect of different amounts of lime used in the preparation of the soap base on the melting point and consistency of the resulting grease. The soap base was analysed in each case to determine the completeness of the saponification. The results show that the soap base made with even some excess of lime, *e.g.* 15.67 parts per 100 of fat (14.08 parts of lime being required theoretically for complete saponification) contained small amounts of free fatty acids and 2 to 3 per cent. of unsaponified fat. A deficiency of lime produces grease of low melting point and excess of lime high melting point products. The highest yield of grease of the most satisfactory appearance is obtained by using slightly more than the theoretical amount of lime. R. W. L. C.

310. Aviation Engine Oils. E. R. Lederer and F. R. Staley. *Oil and Gas J.*, 1929, **27** (44), 158.

The desirable qualitites of aviation engine oils are discussed more particularly in relation to their pour points. The relation between the per centage of wax added to a partly dewaxed paraffin base oil and the A.S.T.M. pour point is shown to be representable by a straight line graph. The effect of added wax on the penetration of a chilled oil has been determined by means of an A.S.T.M. grease penetrometer and curves are given for temperatures

of +10°, zero and —10° F. The three curves are of the same general form. Reduction of the wax content from 0.73 to 0.53 per cent. causes the temperature at which the oil changes from a plastic liquid to a plastic solid to be reduced from +10° to —10° F. The fluidity or the temperature at which the oil ceases to flow and also the effect of added wax on this fluidity has been determined for a number of typical oils in a pressure viscometer under a pressure head of 7 lbs. per square inch. In one instance a Pennsylvanian oil having a pour point of 20° F. showed a better fluidity than an asphalt base oil having a pour point of 5° F. Generally, oils cease to flow under these conditions at temperatures of 20° to 45° F. below their A.S.T.M. pour points. The observed differences with various oils are ascribed to the nature and amount of the wax present in them. R. W. L. C.

311. Flash Vaporisation of Petroleum for Producing Lubricating Oil Cuts. W. A. Peters, Jr., and J. V. Jirasek. *A.P.I. Bull.*, **10**, 45–52.

The purpose of vaporising petroleum is to separate it into fractions according to boiling point and other characteristics, such as viscosity, specific gravity, etc., but as these characteristics are so closely connected with the boiling point, the true boiling point curve of the crude petroleum will give the fundamental data required to solve any problem of vaporising and separating it into fractions.

The typical boiling point curve for Ranger (North Texas) crude is given. The lower and middle parts of the curve are obtained from the data given by laboratory fractionating assay apparatus and the upper part estimated from the analyses of heavy cuts, flash vaporised. at low pressures.

Curves which illustrate the relationships between per cent. distillate and flash distillation temperatures, viscosity and yield of bottoms, and viscosities of the overhead cuts are also given. These curves are in pa t an extension of the work of Leslie and Good (*Ind. and Eng. Chem.*, **19**, 453). A useful rule is stated which is applicable to both single and multiple flash vaporisation, viz., in order to get practically all of a given material out of a residue by flash vaporisation, it is necessary to flash at a temperature 200° F. higher than the boiling point of the material.

It appears that for the purposes of approximate calculations, a large part of the ligher máterial can be regarded as inert gas, so that with suitable corrections, the curves can be used to determine the yield of distillate for a given flash temperature not only from the whole crude, but from a reduced crude with or without steam and at any pressure. As an illustration of the use of this curve, examples are given of different divisions of the typical crude into various sets of products. In each case the flash temperature is read directly or calculated from the curve with, in four cases out of five, excellent agreement between the calculated and the estimated actual flash temperatures. The calculated heat requirements for the various methods of flash vaporisation of Ranger crude show clearly the fuel economy of the vacuum operation and even of the double flash operation without vacuum. It is safe to say that the two-stage operation with the second stage operating under vacuum will always be the more economical method from a fuel standpoint alone. Where the quality of the products is of prime importance, the vacuum unit has a greater advantage in fuel economy. The crude used for these calculations has a relatively low boiling range, so the differences in fuel between vacuum and atmospheric operations are not so great as they would be with a high boiling Pennsylvania crude. Two very interesting curves are added showing the percentage of viscosity remaining after two minutes heating *versus* (*a*) temperature of pipe-still outlet, and (*b*) temperature of vaporiser. These curves show the very marked effect of heating to temperatures of above

about 750° F. even for the short time of two minutes, on the viscosity of heavy distillates. Hence the importance of flashing at as low a temperature as possible. R. W. L. C.

312. Equilibrium Vaporisation of Oils and the Carrying Effect of Light Ends. R. S. Piroomov and G. A. Beiswenger. *A.P.I. Bull.*, **10**, 52–68.

The need for reliable data for dealing with problems involving equilibrium vaporisation is great, as this type of distillation is the underlying principle of pipe-still operation. The present experimental work was undertaken with the purpose of establishing a simple method of predicting the equilibrium flash vaporisation properties of a petroleum stock. The continuous single flash equilibrium data, the true boiling point and the Saybolt 10 per cent. distillations have been determined for twenty petroleum stocks of different boiling ranges.

The apparatus in which the flash vaporisation data were determined is very similar to that used by Leslie and Good and consisted essentially of a preheater coil and an equilibrium vaporising chamber, which can be maintained at a controlled temperature. The results for a number of the typical petroleum stocks are depicted graphically. The following points are brought out in a discussion of the experimental results :

1. The equilibrium flash curves are practically straight lines except at the ends.
2. The slope of the flash curve and its intersection with the true B.P. curve and the 10 per cent. curve depend on the shape of the latter two.
3. The slope of the flash curve and the per cent. off at its intersection with the true B.P. curve is practically the same irrespective of pressure.
4. The flash curves of a crude and of its various bottoms at 760 mm. or at other pressures are all straight lines (except at the ends) and parallel to each other when flash temperatures are plotted against per cent. bottoms on the crude.

An attempt is made to develop an empirical method of predicting the single flash equilibrium curve of any stock at 760 mm. or other pressures once its true boiling point or 10 per cent. curve is available. The equilibrium flash curve being practically a straight line, the slope and a single point on the line would suffice to define it. It was found possible to establish a simple relationship for the slopes and the points of intersection of the flash curve with the T.B.P. or 10 per cent. curve in terms of the slopes and mean boiling points of the two distillation curves. The relation offered holds only for the distillation curves at 760 mm.

The functioning of flash towers is dealt with and a theoretical discussion is added showing the mathematical derivation of equations governing the equilibrium vaporisation of oils.

In order to appreciate the subject matter of this contribution reference to the graphs is necessary, so the original should be consulted.

R. W. L. C.

313. Production of High Viscosity Oils from Mineral Oils and Tar Oils of any Origin. I. G. Farbenind. E.P. 303,776. September 8, 1927.

Oils of high viscosity are produced by subjecting mineral or tar oils, cracked or hydrogenated oils or tars or their fractions, whilst in the coherent liquid phase, to the action of alternating currents of higher frequencies than

500 periods per second, but not usually exceeding 10,000 periods per second. Ionisable substances such as alcohols, bases, phenols, etc., should preferably be excluded from the oil. As an example, a middle fraction of a cracked brown-coal tar oil, free from bases and phenols and having a viscosity of 1·2 degrees Engler, was exposed in the liquid phase to an alternating current of a periodicity of 7000 per second at 12,000 volts under an absolute pressure of 0·01 atmos. After 30 hours, the viscosity had increased to 125 degrees Engler. H. G. S.

314. Cleansing Waste Lubricating and Other Oils. C. E. Fox. E.P. 306,771. February 28, 1929.

The waste oil is effectively cleansed by circulating hot water through it in a downward direction by means of a thermo-syphon principle. The oil is heated in a slow even manner and circulated by convection currents so as to become thoroughly mixed with the water, without sufficient agitation to form an emulsion. Entrained air is liberated from the top surface of the oil, while the circulating hot water is spread over the top surface of the oil. The water sinks through the oil on account of its heavier gravity, carrying with it impurities which are capable of being washed out or precipitated, and which form a layer separating the cleansed oil from the water.

Soda, acid or other cleansing material such as Fuller's earth may be added to the circulating water to assist in the removal of the impurities in the oil. The manner of carrying out the process, with full details of the apparatus employed, are described. H. E. T.

315. Removal of Wax from Hydrocarbon Oils. R. S. Lane, Assr. to Standard Oil Co. U.S.P. 1,706,120. March 19, 1929. Appl. January 2, 1926.

The invention provides a method of removing petrolatum from oils by the admixture of the oil with a diluent containing 35-60 per cent. naphtha, 25–35 per cent. butanol and from 12–35 per cent. acetone, cooling the mixture and separating the wax from the diluted oil by the difference in their specific gravities. S. W. C.

316. Acid Treatment of Lubricating Oils. G. F. Olsen. U.S.P. 1,704,206. October 11, 1926.

Instead of the usual batch agitation with successive amounts of sulphuric acid, the oil to be refined is treated with the necessary amount of sulphuric acid in one operation by forcing the oil through an injector mixer of the Venturi tube type and introducing a thin annular stream of sulphuric acid on to the outer surface of the confined stream of oil. The flow of the combined stream is restricted sufficiently to produce turbulence and very thorough mixing. The temperature of the mixture is controlled to a temperature favourable to decolorisation and unfavourable to the sulphonation of the oil, so that substantial improvement is effected in both the quality and quantity of the treated oil produced. R. W. L. C.

317. Distillation of Lubricating Stock. A. G. Peterken, W. F. Stroud, and R. B. Chillas. U.S.P. 1,710,240. April 14, 1926.

Lubricating oil stock which will leave a residue of at least 20 per cent. when distilled to 650° F. at 10 mm. pressure is diluted with a lower boiling point oil or a naphtha fraction boiling largely below 500° F. The mixture is heated to 700–800° F. by rapid passage through a pipe still and vaporised in a fractionating tower. The presence of the low boiling point constituents has the

effect of lowering the boiling point of the heavy oil and at the same time increasing the velocity and turbulence of the material in the pipe still, so minimising cracking and permitting the production of a small bottom of high viscosity. The desired fractions are obtained in the usual manner as overhead, side draw-offs and bottoms from the fractionating tower. R. W. L. C.

318. Refining of Petroleum. E. B. Phillips and J. G. Stafford. U.S.P. 1,709,149. April 16, 1929. Appl. August 7, 1923.

This invention provides an improved method of refining hydrocarbon oils with sulphuric acid, whereby the objection to the use of acid in the present-day refining practice are overcome. The invention is carried out without the introduction of atmospheric air: the oil being treated with sulphuric acid in a continuous manner, so that an intimate contact between the oil and acid occurs. The oil after the acid treatment is given a clay or Fuller's earth treatment. The apparatus for treating the oil consists of a series of superimposed cells and having rotary baffles carried by a vertical shaft. Leading from the bottom of the treater is an outlet pipe for the oil and acid tar, which are pumped to a settling tank. In the settling tank the acid tar separates out while the treated oil passes through overflow pipes to an agitator containing Fuller's earth. The oil is thoroughly agitated with the Fuller's earth, and is then pumped to a pressure filter, where the treated oil is freed from the earth. Diagrams of the apparatus are given, together with examples illustrating the application of the invention. H. E. T.

319. Renovating Journal Box Oil. T. W. Potter and L. C. McNamara. U.S.P. 1,709,230. April 16th, 1929.

This invention relates to a method of separating finely-divided animal and vegetable fibres, such as cotton and linen waste, from lubricating oils, the invention being intended to be employed for the recovery of oil coming from used journal box waste. The method consists in heating the used oil in a tank closed to the atmosphere, and discharging into a condenser. The oil is heated under a controlled pressure in the tank, until it becomes vaporised without any appreciable cracking occurring. The vaporisation of the oil is assited by introducing steam into the tank containing the oil. The vaporised oil is discharged from the condenser at a temperature above 212° F., by which means it is obtained free from water, and the animal and vegetable fibres, the latter remaining behind in the heating tank. H. E. T.

320. Fuller's Earth Treating Process. R. R. Rosenbaum. E.P. 284,327. January 23, 1928. Conv. (U.S.A.) January 29, 1927.

The invention provides for the treating of Fuller's earth by forcing liquid sulphur dioxide through the Fuller's earth under pressure to flush and dissolve out the oil. When the pressure is released the remaining sulphur dioxide escapes as a gas. S. W. C.

321. Cleansing or Filtering Lubricating Oils. F. R. Sims and B. C. Joy. E.P. 307,547. November 14, 1927.

A magnetic device is described for the removal of para-magnetic particles from lubricating oils. It consists of a magnet surrounded by a cage of diamagnetic character from which the para-magnetic particles can be washed and removed from the magnet. R. W. L. C.

322. Lubricating Oils. Sun Oil Co. E.P. 305.846. March 7, 1928.

By distilling off the lighter fractions from a crude oil a suitable lubricating stock is produced.

Lubricating distillates are distilled from such stock, either with or without admixture with caustic soda or other neutralising solution, by bringing flowing streams of oil distributed over extended surfaces into heat exchange relation with mercury vapour or an equivalent heating agent, while so limiting the temperature of the mercury vapour and the absolute pressure on the oil as to effect the vaporisation of the oil under conditions which minimise cracking.

Access of oxygen is prevented to avoid the production of oxidation products. The lubricating oil vapours are first subjected to reflux condensation under a high vacuum in a tower and the condensate containing the desired fractions is removed and flowed in a shallow stream over an extended surface while subjected to a high vacuum to remove odour-producing gases.

R. L. W. C.

323. Production of Lubricating and Insulating Oils. E.P. 305,553. October 7, 1927.

Substances of the nature of coal, tars, mineral oils and other carbonaceous materials are subjected to destructive hydrogenation and such of the resulting products as are liquid are exposed to the action of high-tension and preferably high-frequency electric currents. For example, brown coal is hydrogenated under a pressure of 200 atmospheres at 450° C. and a fraction of the liquid products which boils up to (circa) 300° C. and does not display any lubricating properties is subjected in the coherent liquid phase to a treatment with a high frequency current of about 10,000 volts and 500 periods. After even a short time, a considerable increase in viscosity is observed. If the treatment be sufficiently prolonged the viscosity can be raised to a multiple of its original value.

R. W. L. C.

324. Mineral Lubricating and Insulating Oils. E.P. 307,649. April 14, 1928.

Lubricating and insulating oils possessing complete freedom from acid, reduced content of tar, stability of colour and resistance to oxidation are produced by treating an appropriate lubricating oil distillate with liquid sulphur dioxide and then subjecting the insoluble portion or refined oil thus obtained to distillation under reduced pressure over alkali with or without the use of steam.

R. W. L. C.

See also Abstracts Nos. 295, 298, 300, 301, 302, 346, 347 and 348.

Special Products.

325. The Paraffin Content of Asphalts for Road Making. H. Burstin. *Petr. Times*, 1929, **21**, 532, 531. (Translated from *Petroleum*.)

It has always been considered one of the basic principles of asphalt road-making that asphalts containing paraffin are not suitable for the purpose. Marcusson states that "Asphalts containing paraffin are liable to become brittle and are therefore little suited for road making." On the other hand, the presence of paraffin in petroleum asphalts was the original cause of natural asphalt being preferred to the artificial product, when asphalt was first used for road making. Technical opinion has been modified to the extent that a small percentage of paraffin in petroleum asphalt (only slightly exceeding

that of natural asphalts) is regarded as innocuous. This is especially so when the paraffin is akin to natural wax and not in the form of crystals. It is not brittle but elastic and pliable and is therefore a component of equal value with the other hydrocarbons of the bitumen, if its percentage is within the limits indicated. Tables are given showing the paraffin content of asphalts from various sources, and from these it will be seen that the paraffin contained in petroleum asphalts varies between 2·2 to 13·5 per cent. of the oily constituents, and roughly 1 to 6 per cent. of the actual asphalts. There is no universally accepted method of determining the percentage of paraffin in asphalts, but there are three main methods of quantitative determination: the cracking method, Schwarz's method and Marcusson's method.

In the cracking method, 30 to 40 gms. of asphalt are weighed into a 100 c.c. retort and distilled dry. The oil distilling over is received into a previously weighed receptacle and the wax content of the distillate determined according to Holde's method and then calculated on the asphalt. The imputation against this test is that part of the wax is destroyed by the high temperature of distillation, but the author states that his experience is that the method gives higher results than the other tests. Schwarz treats 10 gms. of asphalt with 4 c.cs. of concentrated sulphuric acid at a temperature of 180° C., stirring with a thermometer until the odour of the acid has disappeared. 40 gms. of bone charcoal or bleaching powder are then mixed with the carbon residue and the mixture extracted with petroleum ether. The solvent is evaporated from the extract and the paraffin content of the latter determined by Holde's method. Marcusson's method is rather complicated, but is the least open to objection chemically. 20 gms. of asphalt are dissolved in benzol and the solution poured into 400 c.cs. of petroleum ether, a little of the latter being used for rinsing. After standing several hours, the asphaltites will have been precipitated and the solution can be filtered. The filtrate is well shaken three times with 300 c.cs. of concentrated sulphuric acid and then neutralised with caustic soda solution. After washing with water, the bulk of petroleum ether is distilled off and the residue dried to a constant weight. The oleose constituents of the asphalt are then distilled down to coke in a small retort, and the paraffin in the distillate determined by Holde's method. It is shown that using each of the three methods, different results are obtained from the same sample of asphalt. Not even the ratio of percentages obtained by one method from different asphalts agreed with that obtained by another method. This may be explained by the varying chemical composition of the asphalts which react unequally towards heat, concentrated sulphuric acid, etc. Hitherto it has not been emphasised that natural asphalts contain paraffin. Natural asphalts contain up to 0·5 per cent., Californian and Mexican residues up to 1·5 per cent. and Polish and Russian asphalts from 2 to 6 per cent. Two artificial asphalts have been made from Galician petroleum which have an advantage over the natural product in that they have more than 99·5 per cent. of bitumen. It has further been proved that they are more fluid at a warm temperature than other well known asphalts, which enables them to penetrate more easily into the road surface. The author concludes that a paraffin content up to 1 per cent. is entirely innocuous in an asphalt destined for road making. W. H. T.

326. The Chemistry of Bituminous Highway Construction. P. Hubbard. *J.S.C.I.*, 1929, 48, 108 T.

A brief discussion is given of the chemistry of bituminous road construction in connection with the use of petroleum asphalt. Asphalts from various sources will show differences of a chemical nature which, to a greater or smaller extent,

affect the physical character of the product. Slight variations in refining processes may also cause differences in composition which may readily be detected, but to the author's knowledge little evidence has been advanced to show that differences in content of sulphur, paraffin scale, asphaltines, fixed carbon, resins, etc., have any direct significance as applied to service results obtained in highway treatment and construction. Owing to the falling off in importation of Mexican petroleum, the use of South American oil as a source of asphalt has necessitated a change in the methods of preparation and distillation. Batch distillation is not an economical method of refining these oils, and in some cases will not effect as good separation of the asphalt from the heavy oils as will the newer continuous flash coil processes. Prolonged exposure of asphalt in shell stills to a temperature in excess of 315–340° C. will cause reactions which are apt to produce materials of inferior characteristics for certain uses. Where, however, oil or residue is rapidly passed through a long coil at above 370° C., no such injurious effects are produced, particularly where the separation has been conducted under vacuum in the presence of steam. It is quite likely that the judicious use of oxidation or blowing will be found to be the most desirable method of finishing off many of the asphalts produced in the flash coil. With regard to air blowing, it has never been proved definitely under what conditions if at all oxygen is added to the hydrocarbons present in the finished asphalt or if the process is one of pure dehydrogenation. Blowing a poorly-stripped asphalt as compared with one which has been more carefully stripped from the heavy oils, produces products of quite different characteristics, and a promising line of study would lie in ascertaining what chemical changes take place in different portions of the asphalt when they are subjected to the action of air at elevated temperatures. When these reactions are more thoroughly understood, it may be possible to correlate them with the suitability of the asphalt for various commercial purposes. The author has given considerable attention to sheet asphalt paving mixtures, and has developed a physical test for measuring the resistance to displacement of such mixtures when subjected to external forces. The stability test is made on compressed cylindrical briquettes 2 in. in diameter, by placing the briquette in a cylindrical mould and determining the maximum load developed in forcing the mixture through a circular bottom orifice 1½ in. in diameter. The test is made at 60° C. and results are expressed directly as pounds maximum load registered by a dynamometer or spring balance. The results so far obtained indicate no practical difference between asphalts produced from Mid-Continent, Mexican, Colombian, and Venezuelan petroleums, although slightly less stable mixtures were produced with certain Californian petroleum asphalts. With other factors constant, the greatest difference in stability of asphalt paving mixtures has been noted between sands from various sources, even when these sands are brought to the same mesh composition.

It has thus been possible to classify sands relative to their stabilising value in asphalt paving mixtures. Dow has shown that considerable differences exist in the adsorption of different minerals commonly present in paving aggregates. From work that has been carried out, indications point to the fact that whether or not adsorption is directly responsible, those sands with relatively high adsorption values possess more pronounced stabilising characteristics than those with relatively low adsorption values. It is shown that far greater differences in stability of the asphalt paving mixture may be caused by variation in sand and mineral filler and normal variations in proportions of filler and asphalt than in the penetration of the asphalt itself within the ten point limit usually specified. The article concludes with a

brief mention of fluid asphalts for surface treating purposes, their classification as straight residuals or "cut-backs," and the value of "cracking-coil" tars for highway construction purposes. W. H. T.

327. THE CHEMISTRY OF ROAD TARS. H. Mallison. *Petr. Zeit.*, 1929, **25**, 339–403.

The properties of various types of tars are described in detail, in particular German road tar I., anthracene oil tar 50/50 and anthracene oil tar 60/40 blends. These are all designated as good road tars and their properties are given in the following table :—

	Road tar I.	Anthracene oil tar 50/50.	Anthracene oil tar 60/40.
Maximum—			
Specific gravity at 15° C. ..	1·225	1·225	1·225
Water content	1·0% by wt.	1·0% by wt.	1·0% by wt.
Content of other distillates			
Light oils below 170° C. ..	1·0% „	1·0% „	1·0% „
Distillates between 170–270° C.	12·0–24·0% by wt.	1·0–15·0% by wt.	1·0–10·0% by wt.
Distillates between 270–300° C.	4·0–12·0% by wt.	4·0–12·0% by wt.	4·0–12·0% by wt.
Maximum phenol content ..	5.0% by vol.	3·0% by vol.	3·0% by vol.
Maximum naphthalene content	5.0% by wt.	3·0% by wt.	3·0% by wt.
Free carbon	5·0–18·0% by wt.	5·0–18·0% by wt.	5·0–18·0% by wt.
Viscosity (Hutchinson) ..	3·0–15·0 secs.	1·0–15·0 secs.	20·0–80·0 secs.
Pitch content	55–65% by wt.	45–55% by wt.	55–65% by wt.

The pitch content is the residue remaining after distillation of normal pitch of softening point 60–75°C.

Road tar I. differs from the other two tars by its higher content of middle oils causing the viscosity curves of this tar to be a little steeper than those of the other two ; road tar I. is also more volatile on heating. The contents of phenols, and naphthalenes for anthracene oil tars are less than those of road tar I., due to the chemical nature of anthracene oil. Anthracene oil tar 60/40 has a high viscosity on account of its high pitch content.

The estimation of viscosity is carried out in Hutchinson's tar tester at 25° C. Rötger's viscosimeter is used for determinations of special tars at higher temperatures.

The maximum naphthalene content for English road tars is 8 per cent., but only 5 per cent. for German tars. This naphthalene content must be limited because of its high volatility, and yet it acts as a liquefaction medium in decreasing the viscosity of the tar. Graphical illustrations show that when increasing amounts of naphthalene and anthracene oil are added to a viscous road tar, 19 per cent. of anthracene oil and only 10 per cent. naphthalene are necessary to effect a decrease of the original viscosity from 75 to 6 seconds.

The tendency to thicken and dry by evaporation of high-boiling tar constituents is shown by Herrmann's process. The tar is spread thinly on a glass slab kept at 20° and observed from time to time.

"Free carbon" is described as that portion of road tar insoluble in benzol. This carbon is a mixture of carbon with certain asphaltic portions which are soluble in tar but insoluble in benzol. They are determined by the quantity of constituents insoluble in tar-oil or better still aniline-pyridine. The asphaltic substances are not fillers, but they represent a very important constituent of the road-building material.

The effect of these solvents, and of 10 per cent., 15 per cent., and 20 per cent. additions of bitumen respectively on road tar is shown in diagrams in which the tar particle is magnified 270 times, except for 20 per cent. bitumen.

	Dropping point.	Ductility at 10° C.
Tar	41°	Over 100 cms.
„ with 10% bitumen	43°	„ „
„ „ 15% „	41°	70 cms.
„ „ 20% „	42°	16 cms.

The larger black particles present when 20 per cent. bitumen is added to the tar represent a complicated system of various phases. W. S. C.

328. Emulsified Asphalts. E. S. Ross. *J.S.C.I.*, 1929, **48**, 112 T.

Emulsified asphalts may be considered as consisting of three definite media —asphalt, water and emulsifier. The asphalt may consist of 45–75 per cent. of the bulk, water from 25–55 per cent., and the emulsifier from 1–10 per cent. In general only the softer grades of asphalt have been used for emulsification purposes, *e.g.*, materials of softening point 110–160° F., and penetration of 35–200. Though claims have been made for processes using harder materials, such finished emulsified asphalts have not appeared commercially. The origin and refining processes by which asphalts are prepared are of secondary importance only, since suitable emulsions can be prepared from practically all grades of asphalt within the ranges mentioned above, although certain types necessitate changes in methods of processing. Some types of asphalts offer great resistance to emulsification in colloid mills where dispersion is brought about by high speeds, whereas in slow speed paddle mixers first-class emulsions can be prepared with the same asphalts. Again, certain types of asphalt are quite refractory with some of the more common emulsifying agents, but will respond to emulsification quite readily with some of the less used dispersing agents. Correct water conditions are important, and an accurate knowledge of the content and nature of the soluble materials present in the water is necessary. These should be controlled, since the subject of electrolytes is intimately associated with emulsifying processes. It is pointed out that a fundamental knowledge of the colloid state of matter is essential in the study of asphalt emulsions, and the importance of surface tension, viscosity, electrolytes, hydrogen-ion concentration and adsorption are explained with particular reference to these types of emulsions. An asphalt emulsion, to be suitable for road work, must remain liquid without separation for an indefinite period prior to application, and resistance to destruction by freezing is a very important requirement. Further requirements are that it must flow freely under all weather conditions, and must penetrate readily the aggregate surfaces to which it is applied. It must break down soon after application and must then leave a layer of pure asphalt thoroughly covering and bonding to the mineral road aggregate. If the asphalt emulsion is to be used in damp or wet weather, it should be one which will break down without the necessity of drying out. The most successful emulsions break down when exposed to the air in contact with aggregates without drying out, merely by the separation of the asphalt and water due to destruction of the physico-chemical conditions which maintain stability. W. H. T.

329. Mixing or Emulsifying Viscous or Adhering Liquids. O. J. Budtz. E.P. 307,283. March 7, 1929.

This invention relates to an emulsifying apparatus, especially adapted for treating viscous liquids, or for emulsifying these liquids in which only some

of the constituents are adhesive. The apparatus is also capable of forming emulsions of asphalt and tar products in water. Details of the construction and method of working the apparatus are given. H. E. T.

330. Manufacture of Materials from or Comprising Chlorinated Hydrocarbons. A. Carpmael, Assr. to I. G. Farbenind, A.-G. E.P. 309,421. Appl. January 4, 1928.

In order to overcome the irritant action on the skin of those handling chlorinated hydrocarbons of wax-like character intended for use as impregnating media, moulding materials, in electric fuses, etc., there is added thereto a relatively small proportion, 5–20 per cent., of a fat, wax, resin, pitch, artificial or natural rubber, and/or a small quantity of sodium phosphate, calcium oxide, magnesium oxide, pyridine, tetrahydroquinaldine, urea and derivatives, or like substance exerting an alkaline reaction. H. S. G.

331. Preparation of Valuable Compounds from the Refining Wastes of Cracked Products, Particularly Cracked Benzines. Galicyjaskie Towarzystwo Naftowe "Galicja" S. A. W. De Piotrowski and J. Winkler. E.P. 309,718. April 18, 1929. Appl. March 26, 1928.

Waste acid from the refining of cracked products (particularly cracked gasoline) is diluted with water or with dilute alkali and the aqueous and oily layers are treated separately. Thus, gasoline from a Cross plant is refined with 3 per cent. of 88 per cent. sulphuric acid at 15°. The somewhat fluid acid tar is diluted, yielding about 30 per cent. of a mobile oil, *d* 0·830–0·860. The aqueous acid layer is then neutralised by means of waste lye (from the washing of the acid-treated gasoline with 4° Bé soda) and is distilled with superheated steam. The distillate, b.p. 80–150°, is then rectified through a column, yielding alcohols (butyl, amyl, hexyl) amounting to 2–5 per cent. of the acid tar. The greater the content of volatile constituents (b.p. about 50–60°) in the original gasoline, the greater is the yield of alcohols. The higher the refining temperature and the greater the concentration of the acid, the lower is the yield of alcohols. The oily layer from the dilution of the tar is neutralised at 60–70° with 4° Bé. alkali, and is then water-washed and allowed to settle for several hours at 60–70°. The oil is then heated to 110° and steam is blown in. The fraction of b.p. 50–200° is refined with alkali hydroxide and sodium plumbite and is then rectified; the fraction, b.p. 140–200°, flash point not less than 50°, is yellow and possesses a characteristic odour and insecticidal properties. The fraction, b.p. 200–280°, is boiled with a manganese or cobalt siccative (*e.g.*, the fraction is boiled for 24 hours at about 180° with 4–5 per cent. of manganese siccative), yielding a varnish-like product. The residue from the steam distillation is cooled to 140° and is then air-blown, giving asphalt-like products of high elasticity, which, being soluble in gasoline, can be employed as lacquers. W. S. N.

332. Treatment of Bituminous Materials. W. H. Hampton. U.S.P. 1,707,759. April 2, 1929.

The invention is not limited in its application to treatment of any specific bituminous material but finds its greatest utility in the treatment of shale or other oil-bearing rock for the purpose of obtaining valuable hydrocarbons therefrom. It comprises mixing with the shale, a mineral oil having a gravity within the kerosine range, digesting the mixture at a temperature high enough to liquefy contained bitumens and under sufficient pressure to prevent vaporisation of the kerosine and heavier fractions. The volatilised portions are subsequently conducted away and the solid matter separated

from the digested mixture. An apparatus for carrying out the treatment is described and consists of a plurality of digesters together with means for passing the material through them. The last digester of the series is adapted to work under super-atmospheric pressure, and is fitted with means for separating the solid from the bituminous material. A cracking retort is connected to the digesters and arranged to receive vapours therefrom.

W. H. T.

333. Manufacture of Emulsions of Bitumen or Pitch for Road Making. T. M. Hickman. E.P. 308,389. March 28, 1929.

The invention relates to improvements in the manufacture of emulsions of bitumen or pitch for road-making purposes, and has for its object the production of a liquid emulsion which is non-separating, non-fermenting and is capable of being stored indefinitely without deterioration. The invention consists broadly of an emulsifying solution comprising a dilute solution of caustic alkali, alkaline carbonate or of ammonia in which casein is dissolved to which is added a proportion of rosin or its equivalent containing approximately 5 per cent. by weight of acetanilide. It also includes an emulsifying solution of this character wherein a proportion of salicylic acid is used to prevent fermentation of the casein and consequent destruction of the emulsion.

W. H. T.

334. Oxidation of Hydrocarbons. E. C. R. Marks, Assr. to Alox Chemical Corp. E.P. 309,382. Appl. August 6, 1927.

Oxidation is carried out in a vertical or horizontal, stationary or rotary, autoclave, at a pressure of 100–350 lbs. square inch, in the presence of an inciter of oxidation which may be either a catalyst such as manganese stearate or the unsaponifiable non-acidic ketonic bodies produced in a previous reaction. Oxidation is continued until the reaction product is substantially completely emulsifiable, simple carboxylic acids and the acid product insoluble in the petroleum hydrocarbon mixture and in water which is initially produced, are separated from the mixture during the course of the reaction. The final reaction product, which is a mixture of keto-carboxylic acids, hydroxy-carboxylic acids, keto-hydroxy acids and non-acidic unsaponifiable partially oxidised bodies having a ketonic constitution, is treated with an alkali and the unsaponified bodies separated from the saponified constituents.

H. S. G.

335. Bituminous Emulsions. J. A. Montgomerie. E.P. 308,051. March 21, 1929.

A small proportion of fatty acid pitch of vegetable and/or animal oil origin is mixed with pitch and the resulting mixture, when molten, is emulsified with a dilute aqueous alkaline solution.

About 5 per cent. of the fatty acid pitch is added to the pitch or bitumen and 100 parts of this mixture are emulsified with 65 parts of water containing 0·25 parts of caustic potash.

The alkali may be any sodium or potassium salt having an alkaline reaction A small percentage (0·25 per cent.) of soap, such as sodium laurate, may be added to increase the spreading power of the emulsion.

W. L.

336. Chlorinating Hydrocarbons. F. S. Vivas and International Fireproof Products Corporation. E.P. 286,726. March 7, 1929.

Commercial gasoline is first treated with sodium hydroxide solution to saponify the heavy oils and the purified gasoline decanted or drawn off the saponified material, which separates to the bottom. Carbon tetrachloride

(5 gallons), aqueous ammonia (1 gallon) and aluminium or ferric chloride (10 lb.) is added to the purified gasoline (100 gallons). Chlorine is then introduced until the material has d 1·32. The product is washed with alkali. It is non-inflammable and is of use in dry-cleaning and as a solvent in the preparation of paint or varnish. The heavy ends removed from the gasoline by soda treatment are recovered and utilised for lubricating purposes; they may be used in their saponified condition. W. S. N.

See also Abstracts Nos. 275, 279, 307, 309, 349, 350 and 351.

Analysis and Testing.

337. Gas Analysis in Plant Operation. Gas and Gasoline Analysis Committee of California Natural Gasoline Association. *Oil and Gas J.*, 1929, **38**, 151.

This paper discusses how the fundamental analysis of natural gas and gasoline may be utilised for the accurate and absolute determination of the potential production of products from a wet gas, to determine plant and equipment efficiency and to analyse plant performance. It commences with a brief survey of normal methods of analysing natural gas and emphasises the importance of sampling. The physical constants of the hydrocarbons occurring in natural gas being known with considerable accuracy, it is possible to calculate from the analysis of a wet gas its specific gravity, heating value and the liquid equivalents of the separate hydrocarbons, that is the G.P.M.s of propane, iso-butane, n-butane, pentanes and heavier by a very simple computation. Thus, the potential production of any given hydrocarbon or of any specified mixture can be calculated from an analysis of a wet gas.

The analysis of discharge gases from a plant such as absorber discharge, fractionator overhead, accumulator vent gas, etc., indicates the point at which losses of recoverable gasoline occur and the amount of these losses. This method is considered more satisfactory than checking actual production against the potential production as determined from an analysis of the wet gas. The analysis of gasoline is touched on and a table is given illustrating the method of computing from liquid analysis the weights per cent., the mol. fractions, and the partial vapour pressures of the individual hydrocarbons and the sum of the partial vapour pressure yielding the calculated vapour pressure of the gasoline at the temperature used in the calculation. It is possible and desirable when using fundamental analysis to make close material balances for the individual hydrocarbons entering and leaving the equipment and plant in order to determine the dependability and accuracy of the test and to trace the errors involved in it.

The paper closes with a description of the applications of fundamental analysis to plant surveys and to various calculations that have been made possible by it. This description is amplified by tabulated plant surveys and graphs.

The further application of both fundamental analysis and the calculation of composition changes is advised. W. L.

338. The Estimation of Unsaturated Hydrocarbons in Gases. I. Separation by Fractional Distillation. H. S. Davis. *Ind. Eng. Chem.*, Anal. Edn., 1929, **1**, 61–64.

A column is described by means of which it is possible fractionally to distil hydrocarbons, in particular those of low b.p., and especially olefines.

The column consists of 3–5 metres of 5 mm. glass tubing bent into a spiral of 30–40 turns of low pitch. The upright head of the column is surrounded, below the side-arm, by a cup which can be charged with cooling liquid (*e.g.*, acetone). In the cup is suspended a copper gauze on which solid carbon dioxide may be placed, for when acetone-carbon dioxide is employed this arrangements avoids too violent evolution of gaseous carbon dioxide. At the lower end the spiral passes into a small bulb (thus providing a sight for watching the reflux), the lower prolongation of which passes in turn through a paraffin-waxed cork which fits in the neck of a Dewar vessel. The spiral is enclosed in a glass jacket which is highly evacuated and which is silvered inside, unsilvered strips being left on opposite sides. An electrical heating element in the Dewar vessel serves to boil the liquid. The side-arm of the column is connected to a graduated receiver, which is furnished with means for blowing the liquid into another vessel by means of dry air. The receiver is immersed in a Dewar vessel, which, like the cooling cup round the still-head, is fitted with a copper gauze platform.

In operation, the still-head is cooled and the liquid in the Dewar flask is boiled until, after about 30 minutes, there is a steady reflux of liquid. The temperature of the head is then allowed to rise until distillation commences. The fractions of distillate collected in the receiver are blown over into suitable vessels, *e.g.*, thermos flasks, and are stored finally in steel bottles (430 c.c.), of the type used commercially for hydrogen sulphide, fitted with ¼ inch needle valves.

The effectiveness of the column is shown by distilling a mixture of 50 c.c. of benzene, b.p. 79·7–79·8°, f.p. 5·5° and 50 c.c. of toluene, b.p. 110·5° ± 0·05°. The cup surrounding the still-head is charged with water, which is kept at 70–74°. The reflux remains approximately constant at 175 drops per minute. The distillation rate is about 30 drops per minute for the first 35 c.c. and then falls until finally it is 1 drop per minute. The first 50 c.c. boils at 79·7–81·0° and has f.p. 4·8°. The toluene content is 0·6 c.c., benzene 49·4 c.c. The effectiveness, E, is then (49·4 – 0·6)/50, *i.e.* 0·97, and this compares favourably with the results given by a Vigreux column with 14 rows of double points (E = 0·73), and by a Robert column, glass-packed and vacuum-jacketed (E = 0·94).

The first 40 c.c. fractionated from a solution of 40 c.c. of alcohol in 200 c.c. of water contains 94 per cent. of alcohol.

When a mixture of *iso*butylene, b.p. – 6·8°, with an equal volume of Δ^{β}-butylene, b.p + 1·5° to + 1·7°, is distilled, 90 per cent. of the *iso*butylene is recovered in the first 50 per cent. of distillate.

The use of the column in preparing pure hydrocarbons is demonstrated. Propylene, prepared by dehydrating *iso*propyl alcohol with phosphorus pentoxide, has b.p. – 47·3° ± 0·2° at 770 mm.; a fraction, b.p. – 5·8°, is obtained which is soluble in 65 per cent. sulphuric acid and is evidently *iso*butylene derived from tertiary butyl alcohol present in the propyl alcohol apparently to the extent of about 3 per cent. Propylene from the dehydration of *iso*-propyl alcohol by means of active alumina on pumice at 300–325° has b.p. – 47·50 ± 0·3°/764 mm. *Iso*butylene, prepared by heating tertiary butyl alcohol with an equal weight of hydrated oxalic acid, has b.p. – 6·8° ± 0·05° at 760 mm. Δ^{β}-butylene is obtained in 90 per cent. yield by the action of 1·5 vol. glacial phosphoric acid at 117° on *sec.*-*n*-butyl alcohol; it has b.p. 1·5–1·7°. Pure Δ^{α}-butylene is obtained by the action of alcoholic potash on *n*-butyl iodide and has b.p. – 6·9 to – 5·5°.

A light petroleum distillate consisting of field gases and gases from Cross cracking stills, when separated into hydrocarbon groups containing 2, 3, 4 and 5 carbon atoms, is found to contain substantially no ethylene or

ethane, 2·5 per cent. (by weight) of propane, 0·4 per cent. of propylene, substantially no *cyclo*propane, allene or methylacetylene, 4·5 per cent. of *iso*-butylene, 8·7 per cent. of *n*-butylenes and substantially no erythrene.

W. S. N.

339. Analyses of Hydrocarbon Mixtures. P. W. Hill. *Oil and Gas J.*, 1929, **27** (38), 140.

This paper deals with a comparison of the gasoline content obtained by a charcoal test on a sample of wet gas and that calculated from a fractional analyris of the same sample.

The charcoal test gives values within 10 per cent. (plus or minus) of the calculated figure, and the average deviation is about 5 per cent. in favour of the fractionation results.

It is believed that most of the inaccuracy of the charcoal test lies in the distillation, for the amount of condensate collected depends on the relative amounts of the constituents and their vapour pressures. Also, toward the end of the distillation a considerable quantity of fixed gas is given off, probably due to some decomposition. This gas may become saturated with gasoline and so reduce the yield. W. L.

340. A Simple Gasoline Vapour Indicator. H. H. Hall. *A.P.I. Bull.*, 1929, **2** (2), 99.

This paper deals with the general principles of an instrument for detecting the presence of gasoline or other explosive vapours. A sample of vapour from the space under investigation is drawn through a tube, containing a fine platinum wire, which is normally maintained at a bright red heat by an electric current from dry cells enclosed in the instrument. If the sample contains combustible hydrocarbons they will burn catalytically at the surface of the platinum filament, thus raising its temperature and consequently increasing its electrical resistance. This increase of resistance is recorded on an indicator dial which is graduated to show directly the amount of hydrocarbon vapour in the sample. The tube is closed at each end by short brass rod, pierced longitudinally with several very fine holes, to insure that there is no transmission of combustion from the reaction tube to the space being sampled. One end of this tube is attached to a sampling hose by which any part or corner of the space under investigation may be examined and the sample is drawn through by means of an aspirator bulb attached to the other end of the combustion tube. The indicator dial is graduated to show three distinct conditions: (1) mixtures that are safe for men to work in and to use naked lights; (2) mixtures that are probably toxic though not explosive; (3) mixtures that have reached or exceeded the lower explosive limit.

The indicating instrument is a standard millivoltmeter placed across a wheatstone bridge. Current flows through the bridge from the dry battery and is regulated by a rheostat at the proper amount to give the platinum filament the desired temperature. The resistances in the upper arms of the bridge are both made up of platinum filaments, one in the reaction tube and the other in a sealed glass tube full of air. This is done so that a slight inaccuracy in setting the operating current will not cause appreciable error in the indication. When the resistance of the filament in the reaction tube increases the bridge is thrown out of balance so that current flows through the millivoltmeter and deflects the needle over the indicator scale in proportion to the increased resistance.

The instrument is not adapted to the measurement of hydrocarbon vapours in mixtures much above the lower explosion limit, the admission of rich mixtures to the reaction chamber being liable to fuse the platinum filament. W. L.

341. Four Important Requirements in Gasoline Fractionating Column. E. R. Cox. *Oil and Gas J.*, 1929, 27 (38), 140.

The requirements discussed in this article are: a smooth temperature gradient from top to bottom, an adequate number of trays, means for condensing the proper quantity of reflux for the column top and means for providing sufficient ascending vapour at the bottom.

The temperature gradient is rendered smoother by adequate lagging of the column. Thirty trays are considered sufficient, though it is suggested that 50 trays would probably substantially reduce the reflux ratio.

The preferred method of providing the reflux is by pumping back the required amount from the condensate obtained from a primary condenser For heating the bottom, both the built-in heater and the separate reboiler have given satisfaction. The article then discusses the control and disposition of the feed to the column. The feed usually comes from three sources: primary rundown, low pressure condensate and high pressure condensate. To prevent spasmodic working of the column these streams are all confined in one surge tank and fed into the column at one level. They should be heated by an exchanger to a temperature approximating that of the middle trays of the column. W. L.

342. Fractional Distillation Apparatus. W. J. Podbielniak. *Refiner*, 1929, 8 (3), 55.

In the apparatus described, the fractionating column is of Pyrex glass with a cooling chamber at the top and a distilling bulb at the bottom, the latter being electrically heated. Liquid air or any suitable refrigerating agent is blown into a copper cooling vessel immersed in a gasoline bath contained in the cooling chamber. Thermo couples in the vapour space register the temperature of the fractionated vapour and the rate of distillation is controlled by a two-way stopcock, which serves as a flow-regulating valve. A manometer is provided to measure the pressure in the columns and another manometer beyond the regulating stopcock measures the pressure in an evacuated container into which the fractionated vapour passes. The distillate is collected in vapour form in a container of known volume, which is evacuated previous to analysis, and the rising pressure in this container measures accurately the amount of distillate.

The analysis of a gasoline or gas sample consists of (1) entering the sample into a previously evacuated column, the reflux chamber being sufficiently cold to reduce the vapour pressure of the gasoline to atmospheric or to condense the gas to a liquid; (2) running a batch distillation at atmospheric pressure, taking simultaneous readings of the pressure in the receiver and the vapour temperature; (3) plotting a distillation curve (amount of distillate against vapour temperature) from which the percentages of the various hydrocarbons may be calculated.

The fractionating column is surrounded with a silvered vacuum jacket, the silver film being thick enough to improve the thermal insulation about three times and thin enough that visibility through it is retained. The distilling bulb is an integral part of the column and is almost completely surrounded by the vacuum jacket. Heat is applied by an external electric heating element wound on the lower part of the column. Graphs are given to show the accuracy and sensitivity of the apparatus, and it is claimed that efficiency closely approaching 100 per cent. may be attained for components with boiling points far removed. In the case of components boiling close together it is possible to establish accurate cut points by the equal area method. With proper care, duplicate analyses may be made to check within 0·1 per cent.

The apparatus and method of analysis have been found to be of very considerable practical value in controlling the operation of natural gasoline plants, and a table is presented illustrating the use of fundamental analysis in controlling these plants. The apparatus is specially useful in the analysis of liquefied gas products or vent gases from a cracking unit or stabiliser.

W. L.

343. Coated Spiral Fractionating Columns. T. Midgley, Jr. *Ind. Eng. Chem.*, Anal. Edn., 1929, **1**, 86-88.

Satisfactory results are obtained by the use of columns containing a simple or double inner core on which a spiral (or spirals) is wound, the interior of the column being coated with carborundum. W. S. N.

344. Determination of Free Sulphuric Acid in Light Oil Sludge. F. S. Bacon. *Ind. Eng. Chem.*, Anal. Edn., 1929, **1** (2), 89-92.

In this paper an account is recorded of the determination of the free sulphuric acid in light oil sludge by five different methods. The various methods employed were :—

(*a*) Gravimetric determination as barium sulphate.

(*b*) and (*c*).—Titration methods in which the sulphuric acid is titrated with alkali, each under different conditions.

(*d*) The heat of dilution of the sludge with water is determined, and the amount of free sulphuric acid estimated by comparison with data on the heat of dilution of pure sulphuric acid.

(*e*) A method in which the sludge is treated with a boiling solution of aniline in chloroform, with the formation of aniline sulphate. The aniline sulphate is filtered off and analysed either for aniline or sulphuric acid.

All of these methods give reasonable check results on the sludges tested, as is evident from the following table :—

Method.	Sludge I. %	Sludge II. %	Sludge III. %
(*a*) $BaSO_4$	58·4	50·6	59·5
(*b*) Isolation	56·1	49·0	—
(*c*) Titration	58·6	50·0	—
(*d*) Temperature Rise	—	—	60·6
(*e*) Aniline	58·7	50·4	59·2

The methods of determining the amount of free sulphuric acid in sludge obtained from light oils would be of considerable value in refinery operations.

H. E. T.

345. The Rapid Examination of Oils and Essences by the Féry Dispersometer. *Rev. Petrolifere*, 1929, **311**, 311-312.

This article describes the application of a modification of the Féry dispersometer, for the rapid and accurate estimation of the optical dispersion of an oil. The luminous sources employed are : (*a*) a sodium flame giving light of wave length $\lambda_D = 0^{m}{\cdot}5893$ and (*b*) a mercury vapour lamp giving light of wave lengths $\lambda = 0^{m}{\cdot}5461$ (green) and $\lambda^1 = 0^{m}{\cdot}4358$ (indigo). By this means the index of refraction can be measured for light of three different wave lengths. The optical dispersive power of an oil is measured since this constant is practically unaffected by temperature, whereas the index of refraction varies considerably with temperature.

The mathematical theory of the Féry dispersometer is outlined, and it is shown that the total optical dispersion can be calculated from the formula :—

$$\beta = \frac{n\lambda - n_D}{\Delta n}$$

where $n\lambda$ = refractive index for light of wave length λ.

n_D = refractive index for light of wave length D.

Δn = mean refractive index for the light used.

The instrument is capable of being used for estimating the percentages of two oils in a mixture of the two. H. E. T.

346. Carbonisation of Lubricating Oils and Fuel Oils. N. L. Chernojookov. *Ind. Eng. Chem.*, 1929, **4**, 315.

In this paper the author shows that, under the conditions of the "Conradson Carbon Residue Test," the carbonisation of oils depends on the quantities of asphaltenes and tars present in the given oil. Colourless oils and paraffins do not yield appreciable residues of carbon in the Conradson apparatus. It is found that the carbonisation values as obtained in the "Conradson Test" are not a true indication of the carbonisation of oils in actual service. Information concerning the use of oils in service can, however, be obtained by determining the stability of the oils with regard to the formation of sludge. This method is based on the oxidation of oils by oxygen or air under high temperatures and pressures. By the oxidation of refined oils the process of sludge formation may occur in three different ways :—

I. The unsaturated hydrocarbons, tar and sulphur compounds form a sludge, consisting of asphaltenes and carbenes, which may be termed "Sludge of the first type."

II. The saturated hydrocarbons form by oxidation high molecular acids or "Sludge of the second type." This sludge is insoluble in petroleum ether and has an acidity varying between 60 and 110mg. of potassium hydroxide per gram. of acid.

III. A third type of sludge is obtained by the reaction of acids with metals in the presence of water. This reaction proceeds as follows :—

$$2\,R{\cdot}COOH + M(OH)_2 \rightarrow (R{\cdot}COO)_2.\ M + 2H_2O.$$

Different lubricating oils and fuel oils give different quantities of sediment or sludge under identical conditions of oxidation. H. E. T.

347. Testing Automobile Gear Lubricants. C. L. Knopf. *A.P.I. Bull.*, 1929, **2**, 111.

A detailed discussion on the methods of testing automobile gear lubricants is presented in this paper. The requirements of the ideal lubricant for transmission and rear axle gears are stated to be :—

I. It should give a proper lubricating film over a wide range of temperature (from 150° F. down to or below zero).

II. Its consistency at high temperatures should be relatively high.

III. Its consistency at low temperatures should be sufficiently fluid to feed properly to meshing teeth.

IV. It should possess a certain degree of adhesiveness for good pickup and a heavy resisting film.

V. It should have a comparatively long life and, moreover, it must be non-corrosive.

A gear set is described which has been invaluable in the study of a large number of gear lubricants on the market and indispensable in the development of new lubricants. The ordinary pour test is shown to be entirely worthless when used on gear lubricants and semi-solid greases as regards giving accurate data relative to the consistency of an oil at zero degrees. For the purpose of giving accurate data on consistencies at low temperatures, an instrument called the "Viscidometer" has been developed. This instrument measures the consistency or mobility of an oil at zero, and also indicates the ability to feed and non-channel characteristics of the lubricant.

The next instrument described is the "Knopf Adherometer" or "Adhesiveness Tester," which has been designed to yield accurate data on adhesiveness and to determine the relative shifting abilities of gear lubricants. The paper concludes with an account of the use of the "Mac Michael Viscometer," which when properly equipped with efficient temperature control is capable of a high degree of accuracy, measuring the properties essential in a good lubricant. H. E. T.

348. Automatic Viscosimeter. C. M. Larson. *A.P.I. Bull.*, 1929, **2**, 105.

An automatic viscosimeter is described, the chief function of which is to indicate to the motorist when it is necessary to renew his crank-case oil.

The principle and method of operation of the instrument are simple, and, moreover, it is readily installed in the lubricating system of the engine. A small portion of the oil delivered from the pump under a variable pressure is passed through a jet, where it undergoes a dynamic pressure loss proportional to the square of the speed of flow. This pressure drop activates a maximal valve operating within close limits, which holds the pressure drop and oil flow to a constant value. This constant oil flow returns to the zero pressure of the crank-case through a friction tube, whose resistance sets up a pressure head directly proportional to the oil viscosity. A pressure gauge installed in the oil passage ahead of the friction tube can therefore be calibrated in absolute viscosity units or preferably in Saybolt units.

The instrument was calibrated by making two runs on the engine using Asphaltic and Pennsylvania Paraffin Base oils, and plotting the viscosity —temperature and temperature pressure curves. A schematic sectional view of the instrument is given and a mathematical theory is developed.
H. E. T.

349. Determination of the Softening Point of a Pitch or Bitumen. G. M. Green. *J.S.C.I.*, 1929, **48**, 114T.

The author describes a method of determining softening point, which whilst it gives figures one or two degrees above those of Kraemar and Sarnow, is much more easily carried out and the results are reproducible to 1° C. The point of an ordinary sized drawing pin is flattened out into the form of a spear head to a depth of 3mm. The bitumen sample is moulded into a sphere of about 6mm. dia., and this is impaled on to the spear head up to the point where the latter joins the circular shank of the pin. The pin is dropped into a large sized boiling tube fitted with thermometer and stirrer, and containing a solution of potassium iodide saturated at 16° C. This solution has a density which is higher than any bitumen sample, and it has no effect on the bitumen. If the temperature is slowly raised (rate not to exceed ·5° C. per minute near the softening point), there comes a time when the bitumen tails off and floats to the surface. This temperature may be considered to be the softening point of the sample. W. H. T.

350. THE DETERMINATION OF THE MELTING POINT OF PITCHES AND ASPHALTS BY KRAEMAR-SARNOW'S METHOD. D. Holde. *Petr. Zeit.*, 1929, **25**, 411-412.

In this method mercury was first used as the dropping body, but owing to its high density brass rods were used instead of mercury. When brass rods 25 mms. long and 5·5 mms. thick and weighing 5 gms. were utilised as the dropping body for a coal-tar pitch, a softening point of 94-96° was observed when the temperature rise was 1°/minute compared with a value of 82° when mercury was used. At present 5 gm. brass rods 4 mms. in diameter form the dropping medium and the rate of heating is 1°/minute. The difference of the determinations for any pitch is seldom more than 1°. This method has been superseded by the "Ring and Ball Method" for determining the softening points of pitches, bitumens and asphalts, but Holde recommends in preference Kraemar-Sarnow's method. He maintains that in the "Ring and Ball Method" a heating-rate of 5° C/minute is rarely effected, and with hard asphalts the bituminous layer does not adhere strongly to the ring and does not induce a spontaneous drop of the ball. In the Kraemar-Sarnow method the same results can be obtained as with mercury when the brass rods are not greater than 4 mms. in diameter. Application of rods of greater diameter cause the softening point to increase 3-4° compared with mercury with petroleum asphalt and the difference would probably be greater for other pitches. W. S. C.

351. THE DETERMINATION OF WAX IN ASPHALTS AND PITCHES. Luts and Pettai. Report of the Staatliche Brennschiefer Laboratorium. Recd. Mar. 15, 1929.

The paper consists in the main of a comparison of some of the existing methods of estimating the wax content of pitches or asphalts. It is pointed out that there is no reagent which will precipitate paraffin of a certain melting point, while leaving that of lower melting point in solution. The paraffin content is mainly a conventional quantity, which is dependent on the method of analysis, and the latter must be standardised very carefully in order to produce concordant results. In the series of tests described an attempt has been made to compare three methods of wax determination, namely: (1) That of Engler-Holde; (2) the butanone method; and (3) the "English standard" method (I. P. T. ?). For the purpose of the determinations, mixtures of Esthonian shale oil pitch (containing only traces of paraffin) and refined paraffin wax were used. Very brief descriptions of the methods of test are given, together with an account of some of the difficulties experienced in their operation. For example, treatment of the pitch with sulphuric acid is sometimes difficult, owing to the formation of hard lumps which are difficult to crush in the mortar without loss. This is overcome by crushing the mass, while still moist with acid, and afterwards completing the heating on an oil bath. Again, during the cooling of the paraffin wax-solvent mixture, much of the precipitated wax adheres to the walls of the containing vessel like grease and is impossible to remove. The following is a table of the results obtained by each of the three methods:—

Engler-Holde Method.

Wax content of mixture ..	5·2%	Found by analysis ..	4·6%
Wax content of mixture ..	5·4%	Found by analysis ..	4·7%
Wax content of mixture ..	9·2%	Found by analysis ..	8·0%

Butanone Method.

Wax content of mixture ..	2·1%	Found by analysis ..	1·9%
Wax content of mixture ..	5·0%	Found by analysis ..	4·8%
Wax content of mixture ..	10·1%	Found by analysis ..	9·9%

English Standard Method.

Wax content of mixture ..	8·7%	Found by analysis ..	5·5%
Wax content of mixture ..	6·2%	Found by analysis ..	4·1%
Wax content of mixture ..	2·0%	Found by analysis ..	1·5%

The first method is in general inconvenient, and in consequence of the double precipitation entailed and lengthy period of evaporation takes too much time. In the last method the results do not agree with the true paraffin content of the mixtures, and it is in no sense a gravimetric analysis. On the other hand, the determination can be carried out very much quicker than in the case of the other two methods, and gives an approximate idea of the paraffin content of the substance. The butanone method must be given preference over the Engler-Holde method on account of its simplicity and more accurate results. W. H. T.

352. Testing and Maintenance of Fire Foam Solutions. J. B. Rather and D. P. Weld. *A.P.I. Bull.*, 1929, **2** (2), 91.

The paper opens with a general description of the composition of fire foam solutions, and briefly discusses the processes which cause their deterioration.

Methods for estimating the total alkalinity and carbon dioxide content of the sodium bicarbonate solution are given in detail. Determination of the expansion ratio and the stability of the foam are discussed, and methods of test with details of apparatus are given.

A penetration test is also described as being of value in determining the amount of stabilising material required to bring a deteriorated foam back to a good condition and also in comparing stabilising materials. The apparatus used is a standard asphalt penetrometer, the needle being replaced by a waterproofed cork cone with a glass stem adjusted to a weight of seventeen grams. The relationship between penetration and stability of various foams are shown graphically. For reconditioning deteriorated fire foam solutions, the sodium bicarbonate solution should be analysed for total alkalinity and the solution strengthened or diluted to the figure required by formula in use. The corrected bicarbonated solution is then analysed for carbon dioxide content and sufficient carbon dioxide added to bring the gas content to the proper amount. The aluminium sulphate solution is then diluted or strengthened till it shows the correct expansion ratio. Finally the apparent stabiliser content is determined by the penetration test, and the difference between this figure and the stabiliser content, called for by the formula, is added. W. L.

See also Abstracts Nos. 294, 303, 305, 306, 311, 312, 325 and 326.

Refinery Plant.

353. Distilling Apparatus. M. F. De Bajligethy. U.S.P. Re. 17,233. Re. March 12, 1929. Orig. Appl. July 5, 1924.

The invention provides for a still, including an outer shell, a high pressure reaction drum located wholly within the shell; a furnace; an inlet line

passing through said furnace and a housing surrounding the line within the furnace and forming a circulating chamber through which a fused metal may be circulated. The inlet line enters the shell and terminates in the drum, a valve in the line being placed outside the shell; an outlet leading from the drum into the shell with a valve located within the shell, but able to be operated from without, controlling the outlet. Means are connected to the inlet line by which fluid in the drum may be maintained at a higher pressure than the fluid surrounding the drum within the shell. The outlet valve permits the escape of the high pressure fluid in the drum and distributes it throughout the low pressure fluid in the shell and maintains the fluid in the drum under a higher pressure than the fluid in the shell. A vapour line is provided for removing the vapour from the shell. S. W. C.

354. Method of Distilling Petroleum. H. H. Hewetson, Assr. to Standard Oil Development Co. U.S.P. 1,694,259. Dec. 4, 1928. Appl., June 18, 1923.

The invention provides a method of continuously distilling petroleum which comprises maintaining a series of bodies of petroleum at successively higher temperatures, conducting away and condensing the vapours so formed and maintaining a continuously flowing stream of petroleum from each body to that at the next higher temperature. Each stream has a down-flowing and an up-flowing section, and by introducing into said stream a quantity of a liquid having a boiling point lower than the temperature of the stream, a gas is produced by vaporisation in the form of bubbles commingled with the liquid in the upflowing section. S. W. C.

355. Design Should Allow Maintenance of Plant Efficiency. J. A. Millif. *Refiner*, 1929, **8** (4), 69, 72.

The writer starts with the premise that no plant is well designed unless its initial efficiency can be maintained. The design of retorts should be such that the replacement of tubes is comparatively easy. Other important points noted are stack capacity, cost of pumping through tubes and the efficiency under low and high loads.

The efficiency of the retort should not be based on the initial test, but on yearly performance. With regard to heat exchangers it is considered best to sacrifice a little on extreme heat exchanges so that larger flow areas may be employed with a consequent drop in pump pressures and a lower friction drop through the exchangers. It is deemed best to design fractionating towers for low velocities which will allow forcing if and when necessary. Reflux down pipes must have ample capacity and be well distributed.

Medium velocities in a vaporiser are not detrimental provided care is taken to prevent entrainment of residuum with the vapours. Proper insulation is a prime factor in the design of this apparatus. Condensers should be of overcapacity and the water connections by-passed to allow of cleaning without shutting down the unit. W. L.

356. Automatic Pressure Regulation Aids in Natural Gasoline Recovery. J. V. Thomas. *Chem. and Met. Eng.* 1929, **36** (4), 221.

The entire field of pressure regulation as applied to the petroleum industry is divided into six types, namely, reducing regulators, back pressure regulators, gas fuel regulators, pump governors, vacuum regulators and differential or volume control regulators. The design, operation and general application of each of these types is briefly discussed

The primary duty of any regulator is to maintain automatically a given pressure within very close limits. It is the limits within which it is desired to maintain this given pressure that determine whether or not the regulator should be pilot operated. W. L.

See also Abstracts Nos. 285, 288, 292, 316,318, 321, 329 and 352.

Anti-Detonation.

357. High-value Anti-pinking Fuel by the Cracking of Low-temperature Coal-tars. G. Egloff. *Petroleum*, 1929, **25**, 573–577.

The cracking of tar from the low-temperature carbonisation of coal yields (1) cracked gasoline of more than 50 per cent. benzene-equivalent; (2) fuel oil or Diesel oil which may be used as such or may be further cracked to gasoline, gas and coke; (3) tar acids of low b.p., which are suitable for the manufacture of plastics of the bakelite type, or which may be used as insecticides or as wood preservatives: (4) cracked gas of cal. value 1300 B.Th.U. per 1000 cub. ft., containing unsaturated gaseous hydrocarbons, from which ethylene glycol and *iso*propyl alcohol may be prepared; and (5) practically ash-free coke of cal. value 16,000 B.Th.U. per lb., which is useful for metallurgical purposes or for making electrodes, or which, being a smokeless fuel, may be blended with low value coal, or may be converted into water-gas and thence into methanol.

Thus, bituminous coal (1 ton) from West Virginia yields 25 gallons of tar, *d* 1·074, flash point (Cleveland open crucible) 107°, ignition point 127°, viscosity (Saybolt-Furol) 206 sec. at 25°, 0·3 per cent. of water, 10 per cent. volatile to 196°, final b.p. 393° (90 per cent. of distillate), 11·3 per cent. of coke by weight. When cracked at 452° and 7 atm., this material yields 30 per cent. of gasoline containing about 25 per cent. of tar acids or 23 per cent. of acid-free gasoline.

Tar from the low-temperature carbonisation of Ohio-Indiana coal has *d* 1·0794, flash point 54°, ignition point 82°, viscosity 70 sec. at 50°, 27·5 per cent. of tar acids, 1·35 per cent. of tar bases, 10 per cent. volatile to 208°, 78 per cent. to 366° (the final b.p.), and 22 per cent. by weight of coke. At 427° and 7 atm. it yields 34 per cent. of gasoline or 22 per cent. of acid-free gasoline and, per bbl. of tar, 193 lb. of coke and 525 cub. ft. of gas.

Low-temperature carbonisation tar from East Utah coal has *d* 0·9831, flash point 82°, ignition point 104°, viscosity 32 sec. at 50°, 29·0 per cent. of tar acids, 61 per cent. of neutral oil, b.p. 183–394°, 90 per cent. of distillate to 394°, and 11 per cent. by weight of coke. At 435° and 7·7 atm. it yields 24·1 per cent. of gasoline (free from tar acids and bases), *d* 0·7874, b.p. 43–224°, 0·15 per cent. of sulphur, and a benzene equivalent of 65 per cent., together with 17·9 per cent. of fuel or Diesel oil, *d* 0·9315, b.p. 216–399°, and, per bbl. of oil, 120 lb. of coke and 618 cub. ft. of gas.

Neutral oil distillate (free from tar acids and pitch), *d* 0·9478. b.p. 199–374°, 98·5 per cent. volatile at 374°, from the low-temperature carbonisation of bituminous West Virginian coal, at 454° and 17 atm. gives more than 50 per cent. of gasoline of more than 50 per cent. benzene equivalent, and, per bbl. of oil, 85 lb. of ash-free coke, cal. value 16,000 B.Th.U. per lb., and 240 cub. ft. of gas, cal. value nearly 1,300 B.Th U. per 1,000 cub. ft. W. S. N.

358. Service Testing of Petroleum Products. Alois Kromser. *A.P.I. Bull.*, 1929, **10**, 2, 161.

A description is given of some of the equipment used by the Standard Oil Company of California for service testing. Detonation tests of fuels are carried out on a variable compression engine designed in a similar manner to the Ricardo E. 35 engine but having a bore of $3\frac{1}{16}$ in. and a stroke of $4\frac{1}{2}$ in. The compression ratio can be adjusted by raising or lowering the cylinder between the limits of 3 to 1 and 7.5 to 1. Before deciding on this engine, the various methods of evaluating anti-knock value were considered and such systems as the use of fixed compression ratio with throttling or with variable ignition were ruled out, chiefly because they gave no indication of the effect of detonation on power or economy and therefore were unsuitable for the examination of the economic aspects of the question. The engine is coupled to an electric dynamometer. Distilled water is used for cooling to avoid scale formation, and the standard operating temperature is 185° F. outlet. Splash lubrication is used and the oil temperature is maintained constant by means of a cooler. The inlet air flow is measured with an orifice meter coupled to the intake through two surge chambers. Fuel supply is controlled by a positive pump driven by a variable speed transmission gear from the engine and the inlet to the pump is divided to enable different fuels to be tested in succession. The carburetted fuel is heated electrically to maintain constant mixture temperature of 185° F. The intensity of detonation is observed by means of a Midgley indicator with a chrome vanadium diaphragm. This instrument shows a sharp peak on the pressure-time card about $\frac{1}{16}$ in. high with barely audible detonation. The engine is run at 1000 r.p.m. and the ignition advance is set at 20°. Results are expressed in terms of differences of compression ratio required for detonation as compared with a standard fuel. The detonation test is followed by a test to determine the ratio at which the fuel will preignite within 15 mins., and although this test is difficult to duplicate it shows very characteristic differences between fuels. Satisfactory correlation has been obtained between the detonation results from this engine and those from a six-cylinder engine with a 6·5 to 1 ratio controlled by throttling. Lubricating oil tests for determining the influence of viscosity and oiliness on friction are made on a special machine consisting of a hollow journal which can be run at any desired speed. The journal is mounted on ball bearings and loaded with a bearing arranged so that the loading is accurately controlled by hydraulic pressure. The torque transmitted is measured with a Lewis transmission dynamometer. The point of minimum friction at the transition between hydro-dynamic and boundary conditions is clearly shown in the tests.

Transmission greases are tested by measurement of rate of discharge through a pressure viscometer, samples being tested both in the fresh state and after a definite time of stirring. Test engines used for crankcase dilution, examination of effects of sulphur on corrosion, lubricating oil stability, etc., are run under conditions approximating to service conditions as regards starting and stopping, loading, speed and intermittent cooling, etc., and are automatically operated by a suitable arrangement of slow motion cams. R. S.

359. High Compression and Anti-knock Fuels. L. C. Lichty. *S.A.E. Journal*, 1929, **24**, 3, 290.

The economic value of the use of high compression ratios is analysed from the viewpoints of fuel cost, anti-knock cost, decarbonising cost and engine performance.

If thermal efficiency is the only consideration, the cost of power decreases with increase of compression ratio, but at ratios above 5 to 1 the use of a "dope" becomes necessary. The cost of the "dope" determines whether it permits of lower power cost. The author assumes costs and gives diagrams and tables showing maximum permissible increase of price of fuel to justify the use of higher compression ratios than the highest at which undoped fuel can be used. It is shown that with spirit at 30 cents a gallon just satisfactory at a 5 to 1 ratio, it would not be justifiable to change to fuel at 33 cents a gallon even though this enabled a ratio of 6 to 1 to be used. In addition to change of efficiency due to change of ratio the cost of carbon removal is considered. Curves are given based on various gasoline costs and selected "dope" and decarbonising costs showing the net saving over a range of compression ratios. In this respect the saving resulting from the use of a doped fuel is greatest for a low-compression ratio engine.

Engine performance curves at low and high ratios indicate that the effect of increase of ratio is to improve acceleration much more at the higher than at the lower outputs, and it is suggested that increase of ratio should be accompanied by the use of a higher gear ratio to obtain the greatest average advantage under road conditions.

Values from engines with ratios of 4·5 to 1 and 6·5 to 1 are compared. With a suitable alteration of gear ratio this enables extra economy to be obtained with the high ratio up to about half speed, and better acceleration at the maximum speed. The higher ratio engine also runs at a lower speed for a given car speed. R. S.

360. Effect of Anti-knock Materials on the Speed of Initial Uniform Movement of the Flame in Hydrocarbon-air Mixtures. Nagai. *Proc. Imp. Acad.* Japan. Sept. 4, 1928.

The effect of diethyl selenide, tetramethyl tin and tetraethyl lead on the speed of initial uniform movement of the flame in mixtures of air and hydrocarbon vapour were examined in a glass tube 26mm. diameter and 108 cm. long, closed at one end. The mixtures were fired at the opposite and open end. The hydrocarbon used was obtained by the repeated fractional distillation of a gasoline and had a boiling point of 91.6 to 91.7° C. The speeds of initial uniform movement are shown to be distinctly lowered by the addition of anti-knock materials but when the concentration of tetraethyl lead reaches 1 per cent. the curve lowers no further with greater concentrations. With diethyl selenide the same result is reached at about 2 per cent. The results are explained by reference to the theoretical flame propagation temperature of the materials used. When a "dope" is added to a hydrocarbon, it prevents the combustion of the hydrocarbon until the theoretical flame propagation temperature of the dope is reached, at which point the "dope" itself is activated to burn and cannot further raise the combustion temperature of the mixture. R. S.

361. Standardisation of Conditions for Measuring the Detonation Characteristics of Motor Fuels. Stansfield and Thole. *Ind. Eng. Chem.*, 1929. Feb. 1, 1928.

The plant in use at the Anglo Persian Oil Company's research station for the testing of motor spirits is described, including the development of a new design of engine embodying the use of a variable compression head and the Midgley bouncing pin indicator.

The test methods in use on each of three different designs of engine are given and the experimental errors specified. Audibility tests show the greater variations when repeated, the bouncing pin having a sensitivity equivalent

to about .02 of a compression ratio between extreme values on repeat tests. It is shown that samples must be tested unblended, the use of anti-knock material to bring them up to a standard value giving discordant results.

The effect of controllable variables is examined in detail. The most important is fuel feed which must be adjusted to that giving maximum pinking. Selection of suitable spark plugs is necessary to avoid preignition effects. Air inlet temperature and water jacket temperature must be maintained constant but the absolute values do not materially affect comparative results. It is recommended that the ignition setting be kept constant. Tests with various additions of benzole to benzine in each of the three engines show that the increase of highest useful compression ratio varies according to the engine and cannot be expressed either as a difference between ratios or in terms of the ratio between the H.U.C.R.'s. It is concluded that the difference between the results from various laboratories is due mainly to failure properly to control variables on account of lack of appreciation of the effect of these variables; that methods of test vary in accuracy, but that it is possible to obtain results consistent to $\pm$.25 per cent. benzene in heptane; that compression ratio values apply only to the engine on which the tests were made; that large errors may arise if anti-knock values are compared by measuring the amount of anti-knock required to bring samples up to a given standard; that the bouncing pin affords the most accurate method of working.

Pure benzene and n-heptane are suggested as suitable reference standards.

R. S.

See also Abstracts Nos. 281, 284 and 294.

Cracking.

362. Cracking of Light Oils. G. Egloff. *Petr. Zeit.*, 1929, **25**, 507–509.

Contrary to the usually accepted view, a high pressure is not necessary for the production of good benzine in high yield by the cracking of light oils. Thus, an oil of *d* 0·825–0·834, b.p. ·176·7–388·8°, is cracked at 15·4 atm., the temperatures of operation being:—In the liquid 490°, vapour entering dephlegmator 357·2°, reflux liquid or mixtures thereof with fresh oil 426·7°, vapour entering cooling coil 273·9°, oven (above the tubes) 821·1°, oven (below tubes) 579·4° and gases entering furnace stack 399·3°. Pressure distillate of good odour and low sulphur content is obtained in 86 per cent. yield together with 9·5 per cent. of coke and 4.5 per cent. of gas (including loss). The pressure distillate, which has *d* 0·750, b.p. about 29–249·3°, is treated in a continuous treating plant with 2·9 lb of sulphuric acid (66° Bé) per bbl., then with water, soda and water, and is then distilled in a tube still giving 65 per cent. (on the original oil) of benzine, *d* 0·735, b.p. 25·5–218·3°, and fuel oil, *d* 0·8325, b.p. 220–348·3°. W. S. N.

363. Development in Vapour Phase Cracking. C. R. Wagner. *Oil and Gas. J.*, 1929, **27** (49), 46, 149.

The author sketches the early history of vapour phase cracking processes, and discusses the reasons for their failure. The paper is mainly concerned with the Gyro process, which has recently developed on a commercial scale, and has five plants operating and four under construction.

The Gyro process is a low-pressure vapour phase cracking process adapted to the handling of any grade of petroleum oil, and produces a motor fuel

having a high anti-knock value. Briefly, the process consists of pumping cold charging stock into an arrester, wherein it comes into contact with the hot vapours from the converters, and reduces the temperature of the vapours, which enter at about 600° C.

The liquid from the arrester passes out into a hot oil reservoir, and the vapours are discharged into a fractionating tower. The boiling range of the vapours leaving the tower is controlled by a water coil arranged to produce low-pressure steam for use in the process. Enough steam is produced to supply all requirements. The gas produced in the process after separation of the motor spirit is compressed for the recovery of uncondensed light spirit.

The hot-oil reservoir acts as a collector for all the oil which enters the system and has not yet been converted into motor spirit, whilst a hot-oil pump is controlled so that it will maintain a constant level in the hot-oil tank. The quantity of hot oil delivered to the economiser is regulated by an automatic flow meter. The rate of flow is so maintained that the converter is constantly supplied with the correct quantity of vapour. The excess of hot oil is returned to the charging stock tank through heat exchangers or coolers.

The hot oil which passes through the economiser leaves it at about 375° C. and enters the evaporator, where fuel oil drops out as residue and is removed from the system through a cooling coil. Superheated steam is introduced into the system through perforated coils at the bottom of the evaporator, in quantity not exceeding 3 per cent. by weight of the vapours. The vapours and steam pass through a separator to remove entrained liquid, then enter the converter at about 350° C. In passing through the converter, the temperature is raised to about 610° C., and at this temperature cracking takes place. The converter consists of an inlet header and a number of tubes in parallel containing catalytic cores. From the converter the vapours pass into the arrester which has already been mentioned.

The average yields over a period ranging up to 60 days on different gas oils were 52·4 to 55·7 per cent. 220° C. end point distillate, 16·7 to 24·5 per cent. fuel, and 25·2 to 30·0 per cent. gas.

The commercialisation of this process is due to the overcoming of carbon phenomena. Carbon formation was found to be due to lack of control of the cracking reactions or the use of excessive local temperatures. By careful regulation of the time of reaction and by designing the furnace so as to secure efficient heat transfer, the formation of carbon is practically nil. After a thirty days' run charging at 1000 brls. per day, it is doubtful if 100 lbs. of carbon can be collected from all parts of the converter.

Excessive gas production was found to be due to lack of control of the vapour flow in the reaction zone, but more often due to failure to regulate the temperature of the reaction. A variation of 3° C. in the temperature of reaction, all other variables being constant, will cause a wide fluctuation in the proportion between fixed gas and motor spirit.

The refining of vapour phase spirit was difficult, for the product differs entirely from straight run or liquid phase products on account of the highly unsaturated character of the hydrocarbons present in this fuel. It was not an easy matter to remove colouring matter and highly reactive olefins without at the same time reducing the anti-knock value of the spirit. Several methods however are now available which will yield at least 95 per cent. of satisfactory motor fuel. This fuel contains at least 50 per cent. of unsaturated compounds, some of which are responsible for the deposition of gum on standing exposed to air. The complete removal of the unsaturated compounds would reduce the anti-knock value of the spirit.

It has been found that when the spirit is first produced it is free from gum, and while in this condition can be given a certain treatment which inhibits

the formation of gum. Objections are made to the old copper dish test for gum, and reference is made to the method of Smith and Cooke (*Bureau of Mines Reports*, No. 2394 (1922) and No. 2686 (1925).

The motor fuel produced by vapour phase cracking contains no more than traces of paraffins. When analysed by the method of Egloff and Morrell it shows 45 to 50 per cent. olefins, 40 to 45 per cent. aromatics, and the balance naphthenes with paraffins absent. The gas produced in the process contains less than 0·5 per cent. butane and isobutane, hence it seems reasonable to expect only small proportions of the higher paraffins in the motor fuel. By blending straight run spirit with an equal volume of vapour phase cracked spirit, a fuel is produced with an anti-knock value equivalent to 40 to 45 per cent. of benzol. In other words, vapour phase spirit is equivalent to from 80 to 90 per cent. of the value of benzol for blending to produce anti-knock motor spirit. The blended oil is richer in lower boiling fractions than ordinary gasoline. This makes it unnecessary to add natural gasoline as must be done with liquid phase cracked spirit, and thereby increases its value. Also, a remarkable fact is the relatively low sulphur content of vapour phase spirit even when produced from high sulphur crude stock. The sulphur compounds are apparently decomposed and the sulphur appears in the fixed gas as H_2S. The fixed gas varies little in composition excepting as far as H_2S is concerned, and this varies with the sulphur content of the charging stock. An average analysis shows :—

CO_2 – – – H_2S	0·25 per cent.
CO	0·15 ,,
H_2	7·00 ,,
C_2H_4	27·00 ,,
C_3H_6	16·00 ,,
C_4H_8	9·00 ,,
C_4H_6	1·00 ,,
Saturated Hydrocarbons	39·60 ,,

The composition of this gas shows possibilities as an enriching agent in gas manufacture and in the field of chemical manufacture for the production of acetic acid, ethyl alcohol, propylene, isopropyl acetate, acetone, and glycols. The three butanes are all present.

The quantity of raw material in the form of gas may be realised when the production of 5 million cu. ft. of gas in processing 4 thousand barrels of oil is considered. This means :—1,350,000 cu. ft. of ethylene ; 800,000 cu. ft. of propylene ; 450,000 cu. ft. of butenes. R. P.

364. Apparatus for the Treatment of Liquid Hydrocarbons. S. J. M. Auld, A. E. Dunstan and P. H. Herring. U.S.P. 1,711,219. April 30, 1929. Appl., Feb. 11, 1924.

The oil passes through a vertically disposed preheater which may consist of a coil of tubing into a header, from which vertically disposed parallel thermalising tubes conduct the oil into a superimposed reaction chamber. A furnace setting surrounds the three zones of heating, so that heat may be applied to the preheater, thermaliser, or reactor sections, as desired. R. P.

365. Treatment of Hydrocarbons. J. C. Black. U.S.P. 1,706,396. Mar. 26, 1929. Appl., Aug. 12, 1925.

This invention is an improvement on Pat. No. 1,456,419, but employs the same principle of supplying the heat lost by radiation or endothermic reaction in a separately heated element to maintain the cracking reaction at the maximum. The plant consists of a combination of heaters and heat

exchangers. The latter are used to preheat the oil, as a feature of economy, before it passes to the fire heated preliminary coil where the desired heat of decomposition is imparted. From this heater, the oil passes to a second heating coil wherein the temperature of decomposition is maintained.

Simultaneously, a heavy oil is heated in coils placed above the preliminary and secondary cracking coils in the same furnaces.

The secondary coils for light and heavy oils are connected on leaving the furnace and, at a temperature sufficiently high to crack the heavy oil, the mixture is led into a third coil wherein it receives a prolonged heat treatment. The temperature is maintained by supplying sufficient external heat to balance the loss due to radiation or endothermic reaction, in order to obtain a maximum yield of light products.

A pressure is maintained within the heating coils sufficient to prevent vaporization until the oil is discharged into an evaporator and fractionating towers, where the light fractions are separated and the heavier oil returned for further treatment. R. P.

366. Cracking Petroleum. C. R. Downs. U.S.P. 1,708,303. Apr. 9, 1929. Appl. Dec. 1, 1925.

This invention comprises the extension of the principle described in serial No. 692,223, filed Feb. 12, 1924, wherein boiling sulphur is utilized as a method of heating bituminous materials in such a way that distillation takes place with the least amount of superheating and cracking. The extension of the principle is intended to cover the use of boiling sulphur as a means of heating in processes designed for cracking petroleum. Sulphur with a boiling point of 832° F. at sea level can be made to maintain a constant and uniform temperature by regulation of the pressure, and in general the use of absolute pressures of from 9 to 50 lbs. per sq. inch gives a sufficient range for practical purposes. The use of the latent heat of condensation of sulphur vapour as a heat supply renders it possible to obtain high rates of heat flow without excessive temperatures and the production of undesirable products.

The plant consists of a sulphur boiler and a vapour chamber wherein a coil is situated. The oil to be treated is preheated by the waste gases of the furnace, then passed through the coil in the sulphur vapour chamber where sufficient heat is transferred to crack the oil. The required pressure is maintained on the sulphur vapour zone by means of inert gas which is pumped into the system as necessary. R. P.

367. Treating Carbon from Cracking Stills. G. Egloff and H. P. Benner. U.S.P. 1,705,179. Mar. 12, 1929. Appl., Apr. 23, 1921.

The raw stock and reflux oil from the dephlegmator of the plant are pumped together through a cracking coil. The cracked oil passes into one end of a large horizontal cylindrical expansion chamber which is situated outside the heating zone. At the other end of the expansion chamber, a dephlegmator is connected which separates the light fractions from the heavier components. The gas and vapours of the light fractions are cooled and separated whilst the reflux recycles through the process.

At the bottom of the expansion chamber are a series of draw-off pots to deal with the carbon precipitated when expansion takes place. Each of these is fitted with a valve, and beneath the chamber a track is laid over which a waggon may run to receive the carbon from the expansion chamber after the run is completed. At the end of the run the expansion chamber may be one-half to two-thirds full of oil-soaked coke. This is drawn off into the waggon and thence delivered into the hopper of a residue treating still.

The latter is a cylindrical rotary still with stationary ends and has a central heated flue. It also contains steel balls which mechanically break the coke into a fine condition. The distillate is removed through a vapour pipe and condensed whilst the carbon is delivered into a waggon at the stationary end away from the inlet hopper. R. P.

368. Process for Conversion of Petroleum Oils. G. Egloff. U.S.P. 1,707,348. April 2, 1929. Appl. Aug. 16, 1922.

The oil to be treated is pumped into the upper part of a dephlegmator or refluxing tower so that the raw oil serves as a cooling medium for vapours which rise from the cracking zone. The raw oil and higher boiling fractions of the cracked oil collect at the bottom of the dephlegmator and drain downwards through the vapour line which leads from a horizontal reaction chamber to the dephlegmator.

In the centre of this chamber there is a partition, and headers at both ends lead to inclined cracking tubes. Thus the raw oil and reflux flows into one compartment of the reaction vessel, down the header through the cracking tubes and up the other header into the other chamber of the reaction vessel. In the latter compartment vaporisation takes place and the vapours escape to the dephlegmator through the vapour line, counter current to the feed.

Residue and carbon are drawn through a valve placed at the end of the vaporizing compartment away from the centre partition. R. P.

369. Treating Hydrocarbons. G. Egloff, Assr. to Universal Oil Products Co. U.S.P. 1,710,152. April 23, 1929. Appl. Jan. 14, 1920. Renewed Jan. 11, 1928.

Mounted in a furnace is a shell still with the usual manholes and liquid residue draw-off and a vapour line passing into a header connected with a second header by a plurality of relatively small tubes, readily cleanable and replaceable, which lie in the oil in the bottom of the lower part of the still. This header connects with a dephlegmator from which condensed heavy vapours are returned by a reflux line to the still at a point below the liquid level. Uncondensed vapours pass through the dephlegmator to a water condenser and receiver. The process is arranged to be carried out under a uniform pressure of 100 lb. with an oil temperature in the still of 750° F. H. S. G.

370. Cracking Hydrocarbon Oils to Produce Light Oils. G. Egloff and H. P. Benner. U.S.P. 1,710,153. Apr. 23, 1929. Appl. June 28, 1920.

This plant consists of a pair of superimposed horizontal stills connected to each other by conduits so that the vapours from the lower still may pass into the upper still.

A central tube extends through each still. These tubes are connected at one end. An oil burner supplies heat directly into the tube of the lower still, and the heating gases travel through the central tube of the upper still, thence to the stack.

A supply of oil is continually sprayed over the central tube of the lower still and heavy residuum is continuously withdrawn. The vapours from the lower still are superheated in the upper still and pass thence into a dephlegmator. The reflux from the dephlegmator returns to the bulk supply in the lower still whilst the vapours pass overhead into a condenser and separator which remove the gas and pressure distillate from the system. R. P.

371. Production of Liquid and Gaseous Hydrocarbons of Low Boiling Point from Mineral and other Oils and Tars. I. G. Farbenind. E.P. 294,557. Apr. 21, 1927.

Cracking of the specified raw materials is effected by passing their vapours at high velocity over a heating device arranged in a single plane and heated to at least red heat, the linear dimension of the heating element being small in the direction of flow of the vapours. The device may be treated, if necessary, with a blast of gases containing oxygen, or of hydrogen or of such gases or vapours which are free from carbon and are capable of giving off hydrogen. Recycling may be resorted to. H. G. S.

372. Production of Hydrocarbons of Low Boiling Point from those of High Boiling Point. I. G. Farbenind. E.P. 303,761. Sept. 3, 1927, and Feb. 25, 1928.

High-boiling hydrocarbons of any origin are treated with a gaseous halogen hydride in presence of an activated metal or alloy. The contact agents may be activated aluminium, copper, lead, magnesium, iron, cobalt, chromium and the like or alloys thereof, the activation being effected by a solution of a salt of a metal which is less electro-positive than the specified metal or alloy: an anhydrous metal chloride may also be used in conjunction with the activated metal. The reaction is carried out at a temperature exceeding 300° C., whilst the speed of the initial material is controlled so that considerable quantities of hydrocarbons boiling below 70° C. are formed. The raw material and the halogen hydride may also be contacted on the counter-current principle. Nine examples of the method of operation are quoted H. G. S.

373. Production of Hydrocarbons and especially those of Low Boiling Point. J. Y. Johnson, Assr. to I. G. Farbenindistrie Aktiengesellschaft. E.P. 307,946. March 12, 1929.

Suitable catalysts are described for the catalytic production in one stage of hydrocarbons of low boiling range from any kind of coal, tar, mineral oil, their distillation products, residues, etc., by heating to a high temperature and if desired in the presence of hydrogen and preferably under pressure. The catalysts consist of oxides of the metals or metalloids of groups 3 to 7 of the Periodic System deposited in small amounts on the metals aluminium, silver, manganese, chromium or chromium-nickel alloy, which metals are employed in a form presenting a dense coherent surface. The catalysts are applied to the metallic supports in the form of acidified solutions of salts of the catalytic elements. The metals specified for the supports have the following advantages: (1) many metals (*e.g.*, aluminium) themselves possess catalytic properties particularly after being etched and the catalytic oxides are more active when deposited on such etched metals than when used alone or on non-metallic carriers (pumice, charcoal, etc.); (2) these metals do not melt under the conditions of use; (3) they do not give rise to the formation of carbon; (4) the walls of the reaction vessel, pipes, stirring devices, etc., may be formed of these metals and may be coated with the catalysts, an additional quantity of which need not then in certain cases be provided. This is particularly an advantage when initial materials are employed containing ash liable to cause obstruction or to deposit coke when passed through a catalyst layer; (5) owing to the high heat conductivity of these metallic carriers local overheating is less liable to occur. The catalysts described are well adapted to the production of aromatic hydrocarbons from the materials mentioned, the working temperatures being then higher (*e.g.*, 550–650°) than those necessary for the production of benzine.

Thus, an oil consisting of upwards of 80 per cent. of benzine boiling up to 200° is obtained when a mineral oil fraction boiling above 270° is passed with an excess of hydrogen at 450° and 200 atm. through an aluminium-lined vessel containing fine granules of aluminium activated with 1–2 per cent. of uranyl nitrate. The catalyst is prepared by evaporating an aqueous solution of uranyl nitrate, containing about 3 per cent. of hydrochloric acid, in the presence of the aluminium, and then reducing with hydrogen. Similar activity is shown by a catalyst prepared from activated aluminium and boric acid.

An oil, up to 70 per cent. of which boils up to 200°, is obtained by passing a bituminous coal tar oil, b.p. range 300–420°, with hydrogen at 480° and 200 atm., over aluminium gauze, activated by heating with ammonium vanadate and uranyl nitrate in the presence of hydrochloric acid.

Elwerath oil, b.p. above 300°, together with an equal amount of an ash-free middle oil, is forced with the aid of hydrogen through an aluminium-lined vessel at 450°. The vessel contains spiral ribbons of aluminium, previously activated with hydrochloric acid and boric acid. In a single passage, upwards of 40 per cent. of the crude oil passes to benzine and further large proportions to middle oil. If the oil is forced through without the aid of hydrogen the product is obtained in somewhat lower yield and is partly unsaturated. If the temperature is 600° or more, the product contains upwards of 30 per cent. of aromatic hydrocarbons.

In a single passage more than 50 per cent. of benzine is obtained when a middle oil fraction, b.p. 200–350°, from bituminous coal tar is passed with hydrogen at 450° and 200 atm. over silver wool activated by evaporating an aqueous suspension of blue molybdenum oxide containing the wool. Silver wool activated with tungstic acid may also be employed. W. S. N.

374. Cracking of Oils. I. G. Farbenin. Aktien. E.P. 309,227. Apr. 3, 1929. Appl. Oct. 3, 1927.

Excellent yields of valuable liquid hydrocarbons are obtained by cracking tars, mineral oils or other liquid carbonaceous products if carried out in the presence of organic substances containing the radical of a mineral acid, such as aromatic and aliphatic sulphonic acids. Specific examples are benzene sulphonic acid, the residue obtained by evaporation of sulphite cellulose waste liquor, and phenyl borate. Besides the organic substance containing the radical of a mineral acid, other substances which may have a catalytic action may be added such as alkalis, magnesia or zinc oxide.

The additions employed have the advantage that they exert a splitting action on the hydrocarbons under treatment.

The process may be conducted under pressure and, if desired, with an addition of gases not containing substantial amounts of hydrogen. As an example 100 parts of crude oil containing only 4 per cent. of spirit boiling below 180° C. when destructively distilled together with 5 per cent. of benzene sulphonic acid furnish up to 22.5 per cent. of crude product boiling below 180° C. R. P.

375. Treating Fluid Hydrocarbons. F. B. Fretter. U.S.P. 1,707,606. Apr. 2, 1929. Appl. June 18, 1925.

In this process, the oil to be cracked is maintained at a high pressure during its heat treatment, and the treated hydrocarbon oil is suddenly allowed to expand through an outlet such as a Venturi tube, whereby the cracking operation is naturally augmented.

The maximum cracking effect and heat economy are obtained by recycling the treated stock.

The plant consists of a tubular heater which is capable of withstanding a pressure of over 1,000 lbs. per sq. in., at a temperature of 800°F. and upwards. In order to obtain this back pressure on the tubes, a restricted orifice is essential, and in order to provide an orifice suitable for different working conditions, an adjustable nozzle is provided having the features of a velocity tube comprising a gradually constricted neck connecting with a needle valve seat. The needle valve stem is controlled by a screw and hand wheel and has a gland packed to resist the high pressures in use. Against this needle valve the pressure is built up and released into a vaporiser which is open to a tower, at atmospheric pressure. The vapours rise through the tower and are condensed to yield spirit whilst the reflux falls to the bottom of the tower into which raw oil is supplied. The hot mixture of recycle oil and raw oil is fed to the heating coil. As recycling proceeds, the temperature of the heating coil may be advantageously increased. R. P.

376. Art of Cracking Hydrocarbons. Herthel and Pelzer, Assr. to Sinclair Refining Co. U.S.P. 1,703,528. Feb. 26, 1929. Appl. Jan. 17, 1925.

The invention comprises an improved process which assists in protecting the heating surfaces of the cracking still, and which also enables the cracking operation to be continued for a prolonged period before the deposition of carbon begins. The charge of oil is circulated from a bulk supply tank through an externally heated tubular heater and back to the supply tank. The improvement comprises maintaining a bed of material containing uncombined metallic oxides in the bulk supply tank and submerged in the bulk of oil. The material is supported in such a manner as to permit free passage of the still charge. In order to prevent saturation of the still charge with pitch, fresh oil is fed to the pressure still, at the same time drawing off pitch-laden oil therefrom. W. H. T.

377. Cracking of Heavier Hydrocarbon Oils. Herthel and Pelzer, Assr. to Sinclair Refining Co. U.S.P. 1,703,529. Feb. 26, 1929. Appl. May 28, 1925.

The invention relates particularly to improvements in the operation of circulatory pressure still systems in which the oil is heated to a cracking temperature in tubular heaters. A body of Fuller's earth or absorbent material is supported in the still charge so that free passage of oil through the material is permitted. The operation is continued until the pitch content of the still charge builds up at least to saturation. W. H. T.

378. Soaking-Drum Apparatus. H. H. Hewetson, Assr. to Beacon Oil Co. U.S.P. 1,707,520. Apr. 2, 1929. Appl. Mar. 16, 1927.

An oil container adapted to contain its contents at pressures of several hundred pounds and at temperatures of 450°F. to 900°F. is provided with means for keeping the whole interior surface of the container in contact with the oil in its liquid state. A movable extension is placed on the top of the container into which the liquid oil extends during use and means are provided for drawing off oil from the extension and an educt passage communicating with a vapour space above the oil. G. S.

379. Cracking Hydrocarbon Oils. E. W. Isom. U.S.P. 1,708,180. Apr. 9, 1929. Appl. May 21, 1927.

A body of oil is maintained at a cracking temperature by circulating oil from the bottom of the still through a number of vertical tubes thence to the end of the still. The vertical tubes only are subjected to heat from the furnace. The still is maintained about two-thirds full and the process is carried out under pressure.

Vapours pass off through two reflux condensers in series. The heavy reflux condensate from the first returns to the still whilst the reflux from the second is pumped through a separately heated coil and cracked at a higher temperature in the vapour phase. The vapours produced are conducted into the still and discharged through a perforated pipe into the body of the oil. R. P.

380. Art Cracking Hydrocarbons. E. W. Isom. U.S.P. 1,711,351. Apr. 30, 1929. Appl. July 21, 1927.

The invention relates to improvements in the cracking of oils, for the production of anti-knock gasoline in the vapour phase. The process is carried out at temperatures above 480°C.

The oil to be cracked is heated to a high temperature, and the vapours are subjected to a fractionating operation wherein an oil which is free from unsaturated compounds and of similar boiling range to the vapours, is brought into contact with the vapours as a cooling and a condensing agent. The vapours from the fractioning operation are led through a column of absorbent earth or clay before condensation and in this way they are treated whilst fresh from the cracking zone. The heat is thus utilised, and at the same time the cracked or composite product is stabilised by direct treatment without further distillation or blending. R. P.

381. Cracking of Liquid Hydrocarbons. O. D. Lucas and E. L. Lomax. U.S.P. 1,690,416. Nov. 6, 1928. Appl. July 23, 1925.

The process consists of heating the oil to be cracked in a pipe still and passing the heated oils into a conversion chamber in which they are maintained at the cracking temperature and at a pressure of 600 to 1,000 lbs. per sq. in. The lighter oils are drawn off in vapour form into a vapour chamber connected directly to the conversion chamber but at a lower temperature.

The heavier oils are condensed and passed back into the conversion chamber. Light oils pass as vapour to an expansion chamber in which a certain amount of separation takes place and then the vapours pass into a rectification column, whence the motor spirit is led to the treating and condensing apparatus.

The oil from the conversion chamber passes to a coil at the lower end of the rectification column to supply heat. R. P.

382. Method of Cracking Oils. J. R. Mardick. U.S.P. 1,706,629. Mar. 26, 1929. Appl. May 5, 1923.

The essential part of the invention consists in the utilisation of chlorine which has been previously rendered active, *i.e.*, substantially in a nascent condition and at the same time avoiding the presence of objectionable substances such as aluminium compounds which result from the employment of aluminium chloride.

Ordinary chlorine, when activated in the manner specified below, is peculiarly suitable for employment in the production of resin and gum free gasoline and other low boiling fractions from high boiling petroleum residues.

The activating apparatus consists of a vertical metallic cylinder within which an electrode is suspended from an insulated support, and connected to a high potential circuit such as is commonly employed for the production of an electric corona.

Dehydrated and preheated chlorine enters at the bottom of the casing and is discharged at the top through an outlet which conducts the activated chlorine to a still or tower wherein dehydrated and preheated oils are treated. The supply of chlorine, if too rapid, results in the formation of objectionable products, and if too slow, results in low yields of light spirit. A suitable proportion for gas oils appears to be from 0.5 to 1.0 per cent. by weight of the raw oil, while the operating temperature is about 300° C. The product is relatively free from unsaturated hydrocarbons and will not form any appreciable amount of tarry matter when treated with fuming sulphuric acid. In fact, the gasoline obtained requires only an alkaline wash to make a satisfactory product.

The yield ranges from 15 to 45 per cent. of the oil treated. R. P.

383. Treating Hydrocarbon Oil. J. C. Morrell, Assr. to Universal Oil Products Co. U.S.P. 1,710,063. Apr. 23, 1929. Appl. Mar. 31, 1922.

When a cracked distillate, *e.g.*, from Texas, Mexican or Californian oils, is treated with acid, alkali and plumbite and is redistilled, the gasoline distillate may have a high sulphur content. This may be reduced by adding, previous to the distillation, bottoms from a previous re-distillation in such proportion that the gasoline forms 40-50 per cent. of the mixture. It is essential that bottoms from the redistillation of a treated and not an untreated cracked distillate be used. Use is thus made of the tendency of the higher fractions to absorb objectionable sulphur compounds owing to their greater solvent action thereon. W. S. N.

384. Process for Cracking Petroleum. A. Sachenen and M. Tilitscheyew. E.P. 307,105. Mar. 4, 1929. Appl. Dec. 2, 1927.

The process consists of forcing the raw oil at a high pressure through two sets of tubes connected in series and set in the same furnace, heated by waste gases or fuel from the process.

The first (heating) set of tubes is smaller in diameter than the second (reaction) set which is from 4 to 6 times greater in cross sectional area than the heating tubes. The velocity of flow in the heating tubes should not be less than 2 metres per sec. and not less than 0.5 metres per second in the reaction tubes.

The cracked product is led through a reducing valve into an expansion chamber maintained at about atmospheric pressure and in which the carbon is deposited. The heavy residue from the expansion chamber preheats the incoming raw oil.

The vapours from the expansion chamber are led into a rectifying column, and in passing thence flow through tubes which initially preheat the raw oil. The vapours are finally condensed and separated from the gas.

The cracked product from the reaction tube may pass through a coil situated in the bottom section of the rectifying column before entering the expansion chamber.

The reflux from the rectifying tower and, if suitable, the residue from expansion chamber are recycled into the heating and reaction tubes. R. P.

385. Process of Treating Hydrocarbons. A. Schwarz. U.S.P. 1,691,085. Nov. 13, 1929. Appl. Mar. 1, 1924.

This process comprises heating a heavy oil sufficiently to evolve vapour and violently agitating the vapour and oil while being heated to a cracking

temperature by means of an agitator rotating at a speed of from 400 to 600 revolutions per minute, thus forming a vapour-oil emulsion. The apparatus used is a still divided longitudinally by a perforated partition of sheet iron. The agitating stirrer is mounted in the lower compartment while the upper is filled with steel borings or wool.

It is claimed that this process eliminates the dissociation of the oil into fixed carbon and fixed gas and also gives a maximum output of liquids condensable at normal temperatures. W. L.

386. Catalytic Apparatus. The Seldon Company. E.P. 279,818. Feb. 11, 2929. Appl. Oct. 11, 1927.

The invention relates particularly to catalytic apparatus for use in exothermic vapour phase catalytic reactions. The problem of cooling the catalyst is serious when strongly exothermic reactions are carried out. Convertors in which the catalyst is present in relatively large layers have many advantages, but it is very difficult to cool the catalyst uniformly as it tends to become hot in the central zones. In this process, however, an endeavour is made to effect uniform cooling by means of tubes embedded in the catalyst contained in a large layer type converter. The tubes are not uniformly spaced but are packed more closely at the centre of the apparatus. Thus the cold gas or vapour may be passed down the tubes and up through the catalyst and a greater amount of heat abstracted from the centre of the mass of catalyst than from the outer layers.

Five different methods of arranging the tubes and distributing the cooling medium are described. R. P.

387. Apparatus for Converting Hydrocarbons. W. F. Sims, Assr. to Universal Oil Products Ltd. U.S.P. 1,703,550. Feb. 26, 1929. Appl. May 9, 1921.

The invention relates to the conversion of high boiling point hydrocarbons into lighter products such as benzine. The apparatus comprises a heating coil, a reacting chamber and dephlegmator, connected in series. Means are provided for condensing and collecting the vapours issuing from the dephlegmator and also for returning regulated quantities of the distillate to the dephlegmator to be subjected to redistillation while out of contact with vapours undergoing dephlegmation. Regulated quantities of charging oil are supplied to the dephlegmator in order to assist in maintaining a controlled temperature therein. Unvaporised oil can be withdrawn from the reaction chamber and a lower pressure can be maintained on the distillate undergoing redistillation than on the vapours undergoing dephlegmation. W. H. T.

387A. Process of Reducing Ore and Converting Hydrocarbons. W. H. Smith. E.P. 288,193. Aug. 26, 1927. Conv. (U.S.A.), Apr. 4, 1927.

Light hydrocarbons, liquid or gaseous, are obtained from heavy hydrocarbons by introducing the raw material into a continuously moving column of ore at a predetermined point or points relative to a heating zone. The products are drawn off and the ore simultaneously or subsequently reduced. A catalyst may be added to the ore before introduction to the apparatus. G. S.

388. Art of Cracking Hydrocarbons. G. H. Taber, Jr. U.S.P. 1,705,077. Mar. 12, 1929. Appl. Nov. 26, 1929.

All that is claimed for the process is a higher recovery of gasoline from the gas vapour mixture produced in cracking and the plant consists of a number of dephelgmators, coolers, heat exchangers and scrubbers.

The gas vapour mixture from the cracking apparatus escapes to a fractionating column from which light vapour and gas pass overhead and reflux oil flows from the bottom. The vapour and gas proceed through a heat exchanger and cooler, thence to a receiving tank, where the gasoline and gas are separated. The gasoline is allowed to flow to a receiving tank and the gas passes to a scrubbing tower. Cooled reflux from the dephlegmator is supplied to the scrubber and the resultant mixture of light gasoline and reflux oil is returned through the heat exchanger to the dephlegmator. Thus the light spirit recovered from the gas is returned into the finished gasoline receiver. A plant of the Isom type with the necessary additions illustrated shows the complete cycle of operations specified. R. P.

389. Cracking Hydrocarbons. C. P. Tolman. E.P. 299,957. Sept. 17, 1927.

Hydrocarbon vapours are mixed with the vapours of mercury, superheated to a temperature in excess of the cracking temperature of the oil, the heat required to complete the cracking reaction being supplied by the mercury vapours. The process is carried out at a pressure below 50 pounds per square inch. A continuous process is described. H. G. S.

390. Treating Hydrocarbon Oil. J. B. Weaver. U.S.P. 1,708,247. Apr. 9, 1929. Appl. July 22, 1925.

It is claimed for the invention, the combination of a pipe still in which the oil is subjected to heat while in the liquid phase, an expander in which the oil is permitted to expand into the vapour phase, a converter in which the vapours are subjected to further heat action, a tar leg into which the vapours pass, and a fractionating tower. The heavier products are conducted back to the pipe still whilst the motor fuel fractions are collected separately.

The hydrocarbon is pumped into the pipe still and therein raised to a temperature of 700° F. which, however, may be varied according to the character of the raw stock. The heated oils, at a pressure of about 25 lbs. per sq. in., are discharged into the expander. Vaporisation of the lighter fractions takes place and the vapours at about 600° F. enter the converter. The heavy ends are drawn off from the expander chamber.

Just before reaching the converter, the vapours are mixed with steam and the mixture is subjected to a temperature of about 1,100° F. Most of the carbon produced is deposited in cooling from the converter temperature. This discovery is made use of by utilising the tar leg wherein the vapours are brought in contact with the raw stock. Thus the vapours are cooled suddenly to about 450° F., the carbon is deposited in the liquid in the tar leg, and is removed periodically.

By injecting the raw stock into the vapours, light fractions and water are removed before it passes to the pipe still. This also permits of a control of the end point of the spirit from the fractionating tower to which the vapours pass. As a feature of economy, the waste heat from the convertor is used to heat the oil in the pipe still. R. P.

See also Abstracts Nos. 275, 284, 289, 297, 298, 300, 313, 331, 332, 353, 357 and 402.

Oilfield Technology.

391. Unit Operation of Oilfields. D. Comins. *Petr. Times*, 1929, 527, 307.

The article commences with a brief review of the advantages of unit operation of an oilfield, where such a policy is practicable, and emphasises the fact that the ideal development policy for a unit cannot be known unless adequate, consistent and reliable data are available over the whole of the unit. The actual illustrations of unit operation given in the article are those in practice in the Anglo-Persian Oil Company's fields in Persia.

A brief general survey of the Masjid-i-Suleiman unit is given. Local data are collected as a routine throughout the unit. Fluid levels of all gas, oil or water wells, on which measurements are practicable, are recorded monthly, the conditions of measurement being uniform and precise. All gas is bled off from an oil well before its fluid level is measured, and pressures are carefully corrected for instrumental gauge errors. Productions and gas-oil ratios are measured periodically, and the geothermal gradients are also measured in a substantial number of representative wells. Samples of all water and oil shows struck are collected and analysed.

The relation between pressure of oil and its dissolved gas content; the specific gravity of oil under reservoir conditions of pressure; temperature and gas content; the compressibility of high-pressure gas; the relation between temperatures recorded in wells and the structure of the reservoir limestone; the relation between reservoir pressures and the flowing lives of wells; the measurement of the flow in pipelines of oil from which the gas has not been separated; the correlation or recognition of well waters, are all points of interest, and have been the subject of investigation on this and other units. Instances are given showing that data from one unit often provide the key to investigations on other units. Various examples are given showing that the collection and correlation of data will often provide the correct explanation of phenomena in a field which may otherwise be misunderstood with a consequent loss of time and money.

As a result of unit operation it has been possible to collect, interpret and apply the data of the unit to the purpose of conserving capital instead of putting it into the ground; of conserving gas content and rock pressures instead of wasting them; of roughly estimating the reserves and of distributing the accordingly allotted production of the unit in such a way that wells do not prematurely go to gas and do not go to water at all. W. L.

392. Treating Emulsions. C. Fischer and W. T. Reddiah, assrs. to Koutrol Co. U.S.P. 1,710,159. April 23, 1929. Appl., Sept. 2, 1926.

Emulsions are treated with one part in a thousand of a reagent the components of which are :—Sludge layer, mineral oil, aluminium sulphonate and sodium hydroxide, and then maintained at 150–200° F. for a sufficient length of time to initiate coalescence. H. S. G.

393. Dewatering Emulsions. I. Mościcki. U.S.P. 1,710,374. April 23, 1929. Appl., July 31, 1923.

The emulsion to be purified is pumped at high velocity tangentially into a cylindrical heater so that it circulates round steam heated coils. The process is carried out under a pressure in excess of that of the vapours evolved, generally 125–150 lb sq. in. The emulsion passes to a separator of the same dimensions as those of the heater but provided with horizontal baffles. The

oil, freed from impurities and water, flows out of the upper part through a pipe into a coil at the bottom of the heater, where it is cooled by the cold raw emulsion. The velocity of flow is regulated so that every particle of the oil remains in the apparatus for about 6 hours, one-third of the time being in the heater. H. S. G.

394. Treating Emulsified Oil. G. Egloff and H. P. Benner, assrs. to Universal Oil Products Co. U.S.P. 1,710,154. April 23, 1929. Appl., Aug. 21, 1920. Renewed, March 24, 1927.

A cylinder with valve-controlled inlet and outlet carries a movable piston adapted to reciprocate midway between them. Extending through the piston is a plurality of restricted passage ways through which the emulsified oil is forced under sufficient pressure to break the emulsion and allow subsequent separation of oil and water by gravity means. H. S. G.

See also Abstract No. 386.

Geology.

395. Oil Possibilities in Colombia. G. J. Kellner. *Petr. Times*, 1929, 21, 825.

The article is a summary of Colombia's oil possibilities, and gives a short account of the general, tectonic and structural geology of the region. A geological map is given, showing the three main mountainous ranges of the Colombian Andes and the general topography relating to these is described briefly. The general stratigraphy comprises a pre-Cretaceous base of primary rocks overlain by a Cretaceous-Tertiary system. The nucleus of base rocks is formed by phyllites and metamorphosed quartzitic slates, etc., having been penetrated at a later period by neovolcanic or granitic intrusions. The lower Cretaceous-Tertiary beds lie non-conformly on the pre-Cretaceous system, and contain marly sandstones and cemented sedimentary breccia. Capping these beds are the Villeta series consisting of dark coloured shales, limestones, clays and marls interbedded with less important sandstones and quartzites. These beds are taken as the main source of the oil. The next group is known as the Guadalupe series, and is composed of sandstones in square shaped blocks containing upper Cretaceous fossils. Above this is the Guaduas series with interbedded coal seams in the upper horizons, of Eocene age, and representing the horizon for the first oil sands. Diluvial and Quaternary deposits are mainly encountered in river beds, as terraces, mesas or table topped hills. During the pre-Cretaceous age, the beds were affected by folding and subsequent granitic intrusions, and during Eocene and Oligocene times the overlying Cretaceous and Tertiary rocks were severely displaced by a gradual slipping of petroliferous slabs defining to a large extent the "graben" structures of the Magdalena and Cauca rivers. After a period of stabilisation new movements were caused by volcanic eruptions, resulting in the intrusion of dykes and the covering of the "graben" sediments. During the Pliocene age, the sedimentary deposits were again displaced and uplifted by complicated folding and faulting, while in the post-Diluvial period, violent volcanic action covered large areas with ashy products. The foregoing tectonic movements within the petroliferous zones situated along the strike of the graben, influenced the genetic conditions for oil deposition or accumulation and considerably reduced the oil quantities by shrink-

ages in their lateral extensions. The movements destroyed the oil pools with the exception of those situated in or in the neighbourhood of the Mares concession. The oil structures in the Magdalena have been drilled and estimated as non-productive, and thus it would seem logical to locate oil wells in less disturbed localities. The structures on the Pacific Coast of Colombia, where various seeps are reported, seem much more favourable than those mentioned above. W. H. T.

396. Magnetometer Survey of Louisiana. L. Sparagen. *Oil and Gas J.*, 1929, 27, **46**, 103; **48**, 85.

The object of this article is to point out the value of magnetometer surveys in the northern part of Louisiana, where the seismic and geophone methods are not so well adapted. It is shown by means of profiles that in a majority of instances, the known oil-bearing areas are associated with unusual disturbances, and out of the 26 areas examined, 19 occurred in conjunction with magnetic highs. This in itself, of course, gives added importance to the other high magnetic areas, where as yet no oilfields have been found. The very favourable results of the magnetometer obtained in tracing the outline of the Sabine uplift should give added weight to the value of the magnetometer for exploratory detailed work in this area. In conclusion, attention is again called to the general magnetic high areas in northern Lousiana, where a detailed survey should prove of economic value. W. H. T.

397. Physics in Relation to Oil Finding. A. O. Rankine. *Nature*, 1929, 123, 684-686 and 718-720.

The paper gives an account of the seismic method of locating structures likely to be oil-bearing. This method can be applied in rough country, where gravity measurements are too much distorted by surface effects to give reliable indications of underground conditions. It depends not only on the relative densities, but also on the relative elasticities of the rocks encountered or the speeds of propagation of longitudinal mechanical disturbances in these media. Great disturbances of the earth's surface, conveniently in the nature of explosions, are necessary effectively to penetrate to the depths at which oil-bearing structures are frequently found. The necessity for using an explosion involves a difficulty on account of the appreciable time the consequent disturbance of the earth lasts. The solution to this difficulty lies in the existence in practice of another disturbance associated with the lower (higher velocity) medium, but distinct from the reflected disturbance, which may, at a sufficient distance from the explosion, reach the seismograph first. Although small, its time of arrival can be readily recognised since it makes its record on the seismograph before the latter becomes violently disturbed by the direct waves. A simple case is that of two superposed strata, in which the velocities of compressional waves are V_1 and V_2, the latter corresponding to the lower medium and being greater than V_1. On an explosion taking place, three distinct disturbances reach the seismograph. One goes direct to the instrument, one is reflected from the lower medium and arrives later, while the remaining disturbance arrives at the seismograph at a time corresponding to the equivalent path: from the explosion to the lower medium at the critical angle, thence along the junction of the strata, finally travelling at the critical angle up to the instrument. As V_2 (the velocity in the lower stratum) is the higher, it is evident that if the distance from explosion to instrument is sufficiently large, then the total time for transmission may be equal to or less than that for the direct path. If so, its small effect will be recognisable on the seismograph in spite of the

large disturbance which follows afterwards. If a time graph is plotted, with the intervals between the instant of explosion and that of the initial disturbance of the seismograph as ordinates, and the distances between explosion and seismograph as abscissæ, it will display a break where the times of arrival of direct and indirect disturbances are equal. Actually the distance for time equality is related to the depth of interface by the relation

$$\frac{h}{x} = \frac{1-\sin\theta c}{2\cos\theta c}$$

Moreover, the slope of the graph from origin to the position of time equality, which corresponds to the direct disturbance, is proportional to $\frac{1}{V_1}$. The slope from the position of time equality onwards which relates to the indirect disturbance, is proportional to $\frac{1}{V_2}$. Thus, $\sin\theta c = \frac{V_1}{V_2}$, and can be determined. Its insertion in the above equation, together with the value of x (distance for time equality) read from the graph, enables the depth of the interface to be calculated. Owing to its relatively large magnitude, it is possible to recognise on the seismograms the arrival of the direct disturbance even when it reaches the seismograph after the indirect disturbance.

In all cases the procedure has to be the assumption of various possible underground structures until one is found which, by calculation, agrees with the time graphs actually obtained. For this purpose it is frequently necessary to multiply observations by changing the position of the explosion point and the direction of the line of observation. The accumulation of field data over various structures also obviously facilitates the recognition of similar structures in subsequent surveys. Where salt or limestone anticlines are deeply buried, large charges must be exploded, because of the long ranges which must be covered to reach and pass beyond the point of time equality—an essential condition if the depth is to be determined. Consequently, it is economical to multiply the number of seismographs rather than the explosions. For celerity of survey, the seismographs must be readily portable and easily set up in their new positions. W. H. T.

Coal and Shale.

398. Coke as Fuel. T. E. Hurley. *Automobile Engineer*, 1929, 19, 253, 135.

Tests made at H.M. Fuel Research Station with the "Tulloch-Reading" producer are described. The fuels used were charcoal (for which the producer was designed) and low-temperature coke made by carbonising Dalton Main coal in vertical cast iron retorts at 625° C. Power output and flexibility were examined and gas analyses made during the tests. A further prolonged run at constant speed was carried out with coke until choking of the producer led to failure of the engine. It is concluded that low-temperature coke may eventually prove satisfactory as a fuel for certain types of motor vehicle, but that producers designed for charcoal require some alteration to prevent formation of clinker on the refractory lining and to reduce the temperature of the outgoing gas. The tests indicated that coke appeared to be better than charcoal in the quality of gas supplied while idling, in acceleration after short periods of idling and in flexibility generally.

R. S.

399. Recovery of Benzol from Coke Furnace Gas. M. Minot. *Chim. et Ind.* 1929, (2), 252.

The yield of benzol in the actual plant does not exceed 80 per cent., on account of the presence of moisture, impurities such as dust, tar, naphthalene, iron sulphide, cyanide and ferrocyanides, and ammonium thiocyanate which form an emulsion. Moreover, the contaminated oil deposits an insulating layer on the internal walls of the refrigerating coils and so hinders refrigeration. Finally, the yield of benzol decreases gradually as the oil ages and this is due to the decrease in fluidity of the oil by polymerisation at the temperature of overheating of the oil, *i.e.*, 120° C. These deficiencies are remedied by heating the superheating column less strongly, *i.e.*, 105—110°C. The impurities are removed and polymerisation prevented by continuous centrifuging of the oil from the benzene washings. The yield is then raised to 90 per cent. W. S. C.

400. Separation of Acid Constituents from Low-temperature Tar. C. Bunge. E.P. 306,738. March 27, 1928.

The tar or its fractions is emulsified with water or suitable liquor and the emulsion treated with a solvent which, in presence of a considerable amount of water, dissolves the acid constituents. The solvent may be alkali, alcohol, acetone, mixtures of the two latter, and the like. Formation of the emulsion is assisted by adding small quantities of soap. H. G. S.

401. Carbonisation of Coal. G. Cellan-Jones. E.P. 307,811. March 12, 1929.

This is a method of utilising the heat of the waste gases from high-temperature coke ovens for the carbonisation of coal at a lower temperature.

A D-shaped retort of suitable refractory material is placed in the space connecting the combustion flues to the regenerator and extends from the pusher to the coke side of the high-temperature oven. Whether the regenerator is pre-heating incoming cold air or carrying away hot gases, the space surrounding the introduced retort remains practically constant at about 800° C.

When the high-temperature oven is put into operation a charge of low volatile or non-coking coal is fed into the low-temperature retort. Carbonisation takes place and the gases from each low-temperature retort pass into a common main and thence to the suction main of the high-temperature ovens. The semi-coke produced may be utilised to form from 10 to 30 per cent. of the charge for the high-temperature ovens.

The invention is applicable to ovens of the type which reverse across the whole wall length or across the crown of the oven from wall to wall, but not for ovens which reverse for a period on half of one individual wall. W. L.

402. Low-temperature Carbonisation of Coal. W. M. Cranston. E.P. 309,231. August 13, 1928.

A mixture of coal and oil is distilled at a temperature not exceeding 180° C., the residue being then cracked and distilled in a second retort, water or superheated steam being introduced into the second retort to control temperatures and to effect hydrogenation of the unsaturated hydrocarbons. The second retort may be vertical, in which case the temperature at different levels is varied; or it may be horizontal and fitted with stirrers formed preferably of nickel and iron. Distillation in the first retort is effected at pressures slightly less than atmospheric and in the second retort at 20 lb. per sq. in. H. G. S.

403. Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind. E.P. 275,662. Aug. 8, 1927. Conv. (Ger.) Aug. 7, 1926.

The walls of the reaction chamber and of those parts of the plant which are in contact with hot organic reacting materials are kept free from substances which give rise to the formation of methane and the separation of carbon. The hot parts of the apparatus are made of, or are coated with, alloys containing cobalt, molybdenum, tungsten, vanadium or manganese, or their mixtures with, in addition, a considerable percentage of nickel. Again, the walls may contain boron, arsenic, antimony, silicon, bismuth, phosphorus or selenium; or, if the reacting materials are free from sulphur, copper or its alloys may be employed. The metals or alloys should be substantially free from impurities in the boundaries of the crystal grains, and they may be prepared by fusion in vacuo. H. G. S.

404. Production of Hydrocarbons from Coal, Tars, and Mineral Oils. I.G. Farbenind. E.P. 286,680. Sept. 9, 1926. Conv. (Ger.) Sept. 10, 1925.

Carbonaceous materials are destructively hydrogenated in the presence of a catalyst containing an oxide or carbonate of uranium under such conditions as to produce substantial amounts of low boiling hydrocarbons of benzine character. The catalyst may also contain the metals of the eighth group of the periodic system or their compounds. H. G. S.

405. Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind. E.P. 295,947. Aug. 20, 1928.

The destructive hydrogenation of carbonaceous materials at elevated temperatures and at pressures of at least 50 atmospheres is carried out in the presence, as catalysts, of metals of the 5th or 7th group of the periodic system, or compounds of these elements, together with aluminium or compounds thereof. H. G. S.

406. Destructive Hydrogenation of Carbonaceous Materials. I.G. Farbenind. E.P. 295,948. Aug. 20, 1928.

The destructive hydrogenation is carried out in the presence of catalysts consisting of uranium or compounds thereof, together with elements of the 2nd group of the periodic table or their compounds. As an example, crude cresol mixture, together with hydrogen, is passed at 450° C. and under 200 atm. pressure over a catalyst prepared from molecular proportions of zinc oxide and uranyl nitrate, with an addition of 20 per cent. of sugar. The reaction products contain about 50 per cent. of hydrocarbons of low boiling point, in particular of hydroaromatic and aromatic nature. H. G. S.

407. Destructive Hydrogenation of Carbonaceous Materials. I.G. Farbenind. E.P. 295,949. Aug. 20, 1928.

The catalysts used in the process consist of uranium or its compounds together with aluminium or compounds thereof. A large excess of hydrogen in a circulatory system is preferably employed, the pressure being in excess of 50 atm., whilst the parts of the plant which are in contact with the hot reacting materials should be made of or coated with highly alloyed steels and the like, *i.e.*, iron or other materials which give rise to methane, with separation of carbon, should be avoided. H. G. S.

408. PRODUCTION OF LIQUID AND OTHER HYDROCARBONS BY DESTRUCTIVE HYDROGENATION. I.G. Farbenind. E.P. 299,020. Aug. 8, 1927. Conv. (Ger.) Aug. 7, 1926.

The destructive hydrogenation of carbonaceous materials is carried out in a current of gas containing a large excess of hydrogen and in the presence of catalysts containing the metalloids boron, phosphorus, arsenic, selenium or tellurium either in the free or combined state. H. G. S.

409. PRODUCTION OF LIQUID AND OTHER HYDROCARBONS BY DESTRUCTIVE HYDROGENATION OF CARBONACEOUS MATERIALS. I.G. Farbenind. E.P. 299,021. Aug. 8, 1927. Conv. (Ger.) Aug. 7, 1926.

Carbonaceous materials are hydrogenated at elevated temperatures and pressures exceeding 50 atmos., in the presence of catalysts containing halogens in the free or combined state and operating with a gas containing a large excess of hydrogen. The catalysts may, for example, contain hydrofluoric or hydrochloric acid or salts thereof, *e.g.*, calcium fluoride, molybdenum trioxide, with an addition of 10 per cent. of aluminium or cadmium chloride. H. G. S.

410. TREATMENT OF COAL AND OTHER SOLID CARBONACEOUS MATERIALS FOR PRODUCTION OF LIQUID HYDROCARBONS AND OTHER ORGANIC SUBSTANCES. I. G. Farbenind. E.P. 308,633. Dec. 21, 1927. Addition to E.P. 282,691. Conv.(Ger.), Dec. 22, 1926.

The invention has reference to the pre-treatment of solid carbonaceous materials prior to their conversion by destructive hydrogenation and consists in subjecting them to heat and pressure without the addition of organic solvents and of gases containing a substantial amount of hydrogen. The treatment is carried out at temperatures higher than are employed in briquetting, but below the coking point of the raw material, and may be effected in the presence of a substance having an alkaline reaction, *e.g.*, a sulphide of a strongly basic metal, or other substances exhibiting catalytic properties, *e.g.*, iodine or other non-metals. Water may also be present during the treatment, whilst if pressures below 75 atmos. are employed, organic solvents of low boiling range may be added to the raw material. As an example, brown-coal is made into a paste with water and 5 to 10 per cent. of sodium sulphide and heated to 360°C. and under 200 atmos. pressure in an apparatus constructed of steel containing a high percentage of nickel and chromium. About 50 per cent. of the coal substance is liquefied, practically without formation of gas. The liquid product is then separated and destructively hydrogenated in presence of a molybdenum-chromium catalyst to give a product containing about 25 per cent. of benzine, the remainder boiling up to 350°C. H. G. S.

411.—DESTRUCTIVE HYDROGENATION OF CARBONACEOUS MATERIALS. I. G. Farbenind. E.P. 309,228. Oct. 3, 1927 (Divided out of No. 302,941).

Destructive hydrogenation of coal, tars, mineral oils, etc., is effected in presence of such organic substances as contain the radical of a mineral acid in the molecule and which, when admixed with water, lower the surface tension thereof. The substances mentioned are aromatic and aliphatic sulphonic acids or their derivatives or substitution products, sulphonic acids of aromatic or hydroaromatic hydrocarbons sulphonated in the nucleus and containing at least one alkyl group in the nucleus. Alkalis, magnesia or zinc oxide, may also be added. H. G. S.

412.—Extraction of Oils from Carbonaceous Materials by Means of Solvents Under Pressure. J. Y. Johnson. (Assr. to I. G. Farben. Akt.) E.P. 309,229. April 3, 1929.

This invention relates to the extraction of oils from carbonaceous materials by means of liquid organic solvents under pressure. It consists of extracting materials such as coal, tars, mineral oils, etc., with liquids which are capable of acting in the presence of water as wetting and emulsifying agents. As an example, brown coal is stirred up with tetrahydronaphthaline with the addition of .05 per cent. of sodium isopropylnaphthalene-sulphonate, and heated to 350°C under pressure. 75 to 80 per cent. of the coal is converted into products soluble in benzine, while the insoluble residue consists of undissolved coal and ash. Without the use of the wetting agent, the yield is considerably lower. W. H. T.

413.—Low-Temperature Carbonisation and Apparatus Therefor. I. G. Farbenind. E.P. 309,253. Oct. 26, 1928. Addn. to 301,975, Sept. 19, 1927.

The fuel to be carbonised is pre-dried by blowing through it hot scavenging gases at such a rate that the fuel is maintained in a state of active movement on its bed so that it presents the appearance of an ebullient liquid. The material in a dried and pre-heated state is then introduced into the low-temperature carbonisation chamber. H. G. S.

414.—Obtaining Light Hydrocarbons. Florentin, Kling and Matignon. E.P. 279,488. Feb. 20, 1929.

The invention relates to the production of light hydrocarbons from tars containing poly-aromatic nuclei and phenolic substances, by the hydrogenation of both these substances in a single operation. The process consists of treating these complex organic compounds with a metal oxide which acts as a dehydrating catalyst, such as thoria, together with a metal halide (substitutive catalyst), in the presence of hydrogen under pressure. Temperatures between 350° and 480°C are used, the process being carried out in two successive stages at temperatures most favourable to the action of each kind of catalyst. W. H. T.

415. Hydrogenation of Coal, Tars, Mineral Oils, &c. K. Gordon and Imperial Chem. Ind. E.P. 309.239. Jan. 14, 1928.

The process consists in acting upon carbonaceous materials with hydrogen at elevated temperatures and at a pressure of at least 1,000 atmos. Alternatively, the material may first be treated at pressures below 1,000 atmos. and subsequently at over 1,000 atmos. The preliminary treatment at the lower pressure may be effected with circulation of hydrogen, the second stage being carried out with a single passage of hydrogen. H. G. S.

416.—Hydrogenation of Coal, Tars, Mineral Oils and other Carbonaceous Materials. Imperial Chem. Ind. and K. Gordon. E.P. 309,095. Dec. 2, 1927.

The main difficulties experienced in the hydrogenation of carbonaceous materials are carbon deposition and production of large volumes of uncondensable gas. These are due to the slow rate at which the hydrogenation reaction occurs and may be obviated by the use of much higher pressures

than is customary, *e.g.*, 300 to 500 atmos. or over, and preferably 800 to 1,000 atmos., with or without the employment of catalysts. A temperature of 420°C. appears to be the optimum but the process may be carried out at 450°-500°C. under a hydrogen pressure of 500-1,000 atmos. H. G. S.

417. Gas Purification. The Koppers Co. E.P. 283,948. March 18, 1929.

This patent relates to the purification of coke-oven gas, coal gas and similar fuel gases from hydrogen sulphide and analogous acidic impurities by an alkaline solution such as sodium carbonate, and also the regeneration of the spent alkali liquor by a portion of the purified gas.

The foul gas is cooled to about 77° F. and is washed in countercurrent with a 1 to 3 per cent. solution of sodium carbonate. The washing takes place in an absorber fitted with any suitable contact device.

The regeneration of the fouled sodium carbonate solution is accomplished by treating it with about 35 to 40 per cent. of the pure gas. The spent liquor is heated, by waste steam or otherwise, to about 185° F. and is passed downward through a regenerator in countercurrent to the rising gas. The regenerator is preferably of the mechanical rotor type.

Alkali is added to the regenerated liquor to make up for mechanical losses, formation of fixed salts, etc. The exit gas from the regenerator is cooled by direct contact with water and is used for fuel as desired. W. L.

418. Removal of Phenols from Tars or Tar Oils. J. Karpati and M. G. Hubsch. E.P. 283,569. Nov. 28, 1927. Conv. (Hungary), Jan. 15, 1927.

The process consists in treating tar oils under moderate super-pressure and at temperatures exceeding 100° C. with a solvent for phenols diluted with water, removing the resulting phenol solution and cooling and releasing the pressure whereby the phenols and phenol solvent separate into two layers, the latter being available for re-use. The treatment is effected at 1·5 to 6 atmos. at temperatures of 100 to 150° C., the solvents employed being alcohols, *e.g.*, methyl-, ethyl-, or propyl-alcohol, or acetone, formaldehyde, acetaldehyde and the like, diluted with about 4 times the volume of water. A mineral salt may also be used in conjunction with the solvent. The process may be continuous or may be applied for the separation of individual phenols.

H. G. S.

419. Distillation and Utilisation of Carbonaceous Materials and to the Manufacture of Coke, Briquettes and Solid Fuel Compositions. B. Laing and H. Neilsen. E.P. 307,366. Oct. 4, 1927.

A fuel for use in metallurgical purposes includes the solid residue from the distillation of raw solid carbonaceous material by means of heating gases, the magnitude of which is such as to lower the pressure of the volatilisable oils present so that they are evaporated at temperatures below normal. The solid residue may be that resulting from the processes claimed in E.P. 276,407, 287,037 or 287,381. G. S.

420. Hydrogenation of Coal. C. H. Lander, F. S. Sinnatt and J. G. King. E.P. 306,564. Sept. 17, 1927.

The invention relates to the pre-treatment of coal to render it more amenable to destructive hydrogenation. This pre-treatment may consist in removing fusain and/or durain from the coal by hand picking or, after crushing, by any of the known means of eliminating the finer material which contains the bulk of the fusain. Alternatively or additionally the coal may undergo

treatment to modify its coking or caking properties, which alteration can be brought about by a preliminary hydrogenation, by the action of oxidising agents, by heating at above or below atmospheric pressure, or by the action of steam or the vapours of hydrocarbons or other bodies which are not oxidising agents. Raw materials pre-treated by different means may be blended to give raw material which is subsequently destructively hydrogenated.

H. G. S.

421. Distillation of Solid Carbonaceous or Oil-Bearing Materials. H. Nielsen and B. Laing. E.P. 306,654. Feb. 28, 1929. Appl. Dec. 23, 1927.

If the vapours produced during the distillation of solid carbonaceous substances are subjected to excessive temperatures, prior to condensation, the yield is diminished and the oils are poor in quality, particularly when distilling Torbanite or Shales rich in oil. This effect may be produced by the vapours being brought into cooler temperature zones, resulting in partial condensation, followed by re-evaporation due to the fact that the raw material is continuously carried into hotter zones.

In this process, these effects are obviated by preheating the raw material before it enters the distillation retort. Distillation is completed at a temperature above that at which the heaviest oil fractions are liable to condense. The preheating temperature varies from 100° C. to 250° C., according to the volume of gas passing through the retort and the temperature is in inverse proportion to the quantity of oxygen containing constituents present in the raw material.

Distillation is carried out by means of a controlled volume of an inert heated gas. Where the yield of oil is of the order of 20 gallons per ton, the material is preheated to a temperature of approximately 200° to 250° C. Precautions are taken to maintain throughout the length of the retort and dust extractor, etc., a temperature which does not fall below 100°—150° C. until actual condensation of the oil vapours is desired. The hot gas used for distillation is also used for preheating, after cooling or heating. R. P.

422. Dissolving Coal and Product Produced thereby. Trent Process Corpn. E.P. 277,659. Sept. 12, 1927. Conv. (U.S.A.) Sept. 14, 1926.

Bituminous coal in a state of fine subdivision is mixed with a liquid hydrocarbon, *e.g.*, petroleum and the mixture heated under pressure and to a temperature sufficient to produce softening of the coal. The treatment is continued until substantially all liquefiable constituents of the coal have become reduced to liquid form, and have dissolved in or become miscible with the oil, whilst the remaining solid carbonaceous matter enters into and remains in suspension. The mixture is agitated during treatment. The claims embrace the fuels actually prepared by the above means. H. G. S.

ABSTRACTS.

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General.

423. PROCESS FOR THE PURIFICATION OF DETERGENT SOLUTIONS. G. B. Boon. U.S.P. 1,711,829. May 7, 1929. Appl. Jan. 16, 1928.

This invention relates to a new method for purifying detergent solutions, such as gasoline or other solvents which are commonly used in dry cleaning establishments. The solvent to be purified is introduced by downward flow beneath the surface of a solution of caustic soda and is then withdrawn from above the caustic solution. The solvent is next introduced beneath the surface of an aqueous body and then passed through solid calcium chloride, the cleansed solvent being discharged at a point above the solid chloride.

H. E. T.

424. TREATMENT OF SPENT CLAYS. R. A. Dunham, Assr. to Union Oil Co. U.S.P. 1,694,971. Dec. 11, 1928. Appl. Oct. 30, 1926.

The process provides a method for de-oiling spent clays from oil-treating processes comprising bringing a predetermined amount of water into intimate contact with the oil containing spent clay, the water being introduced gradually so that substantially all of it is adsorbed promptly upon introduction, agitating the mass to insure such intimate contact, and continuing the water addition until the mass assumes a stiff homogeneous plastic condition, then removing the oil separated by this procedure from the water and clay mass which forms.

S. W. C.

425. CONTROL IN CONTINUOUS DISTILLATION. C. E. Mason. *Oil and Gas J.*, 1929, **27** (42), 153, 154, 157.

Physical specifications of petroleum products depend on the combined results of innumerable physical reactions in the process of fractionation. For instance, the end-point of a product from a bubble tower will result from the presence of certain high boiling-point hydrocarbons in the vapour state at the top of the tower. It is assumed that this depends on the temperature at that point, but a number of other factors are involved. Pressure variations, although small, are a factor of this specification, and the law of partial pressures is also applicable. Using open spray reflux, the end-point is also a function of the percentage of recycle stock, which is a function of the amount of heat which must be removed in the top trays. The heat to be removed in the top trays is a function of the number of trays, the temperature potential between top and bottom, and the amount and velocity of the vapours. The temperature control of outlet vapours is, therefore, only a compensating control and it is necessary to consider reactions in the pipe— or shell-still.

N

The fundamental principle of the pipe-still is high velocity and a uniform increase in temperature without danger of heating. It would seem logical to control pipe-still outlet temperature by adjusting furnace temperature to compensate for changes in velocity, but the outlet temperature is the result of conditions existing in the still some minutes before. A condition of uniform charging rate is therefore essential to balance operating conditions in the pipe-still, the bubble tower and eventually the end-point. It may be said that the velocity is directly proportional to the amount of vapours generated and inversely proportional to the time of contact. Pressure drop is more nearly proportional to the square of the velocity. Any variation in the rate of flow is best measured by an orifice meter in the charging line and the automatic control of the differential pressure across an orifice plate offers the best means of rate of flow control.

The application of automatic flow control results in a uniform amount of vapours and percentage extraction. This results in uniform supply of bubble tower bottoms and a constant liquid level at the bottom of the tower will assure delivery to the next still at a uniform rate. G. S.

426. Lubrication Vital for Wire Rope. C. D. Meals. *Oil and Gas J.*, 28, 6–40 (53–54).

The rope usually utilised has a hemp or wire rope centre with a core of hempen fibre. Its life depends on the lubrication of the hemp core, and the quality of the lubricant used depends on the type of wire rope. There are five general types of wire ropes :—

1. Slow speed ropes operating under heavy loads, *e.g.*, those used for derricks, dredges, cranes, etc., for which a straight petroleum product with a viscosity of 900–1,100 seconds at 210°F. is suitable and is best applied when hot.

2. High speed hoisting ropes, *e.g.*, mine-shaft ropes for which a lubricant of low viscosity (350–500 seconds at 100°F) is suitable.

3. Haulage and slope ropes run at high speeds and under heavy loads, *e.g.*, furnace hoists, derrick falls, coal mine slope and shaft ropes for which a mineral oil lubricant with a viscosity of 500–800 seconds at 210°F is appropriate.

4. General haulage ropes and ropes of lighter duty than (3) for which a mineral oil having a viscosity of 200–300 seconds at 210°F is recommended.

5. Stationary ropes exposed to moisture, corrosive fumes, or spray, etc., for which a straight petroleum product with a viscosity of 900–1100 seconds at 210°F is recommended.

It is usually found that the greater the speed of the rope, the lower should be the viscosity of the lubricant used on the rope ; it is best to use thicker, semi-plastic lubricants in a hot thinned condition, and if applied cold the lubricant should be chemically neutral.

The method of lubrication varies according to whether the rope is vertical or inclined ; but in any case it is not advisable to remove the rope from service while lubricating. The frequency of application of the lubricant depends on the service conditions. W. S. C.

427. Process For Reclaiming Distillation Residues. R. E. Powell, Assr. to Standard Oil Development Co. U.S.P. 1,694,280. Dec. 4, 1928. Appl. Feb. 25, 1928.

The patent provides a process of removing naphthenates from caustic soda bottoms, which comprises charging the bottoms while hot in a still, adding relatively cool gas oil in amount sufficient to flux the bottoms, heating the

still to maintain the temperature of its contents, injecting sufficient steam to mix the bottoms and gas oil, discharging the fluxed residue upon the surface of a gently moving stream of water, retarding the flow, allowing stratification to take place and separately withdrawing the oil and the water containing dissolved sodium naphthenates. S. W. C.

428. Effect of Cathode Rays on Hydrocarbon Oils and on Paper. C. S. Schoepfle and L. H. Connell. *Ind. Eng. Chem.*, Vol. 21, **6**, 529–537.

This paper deals with the chemical phases of the deterioration of cable during use. The original problem was the study of the reaction resulting in the formation of wax from oils. Samples were subjected to the action of a high electric stress for varying periods of time. A fresh sample of vaseline became a mass of wax after two and a half days, while the same sample which had been carefully vacuum dried, remained unchanged after two weeks' exposure to the electric discharge. When an oil through which oxygen was bubbled was subjected to the action of the electric stress it rapidly changed in colour and became rancid; no solid material corresponding with wax was produced, and hence no indication was obtained that the deterioration corresponded in any way with that found in cables.

A review of the literature is given on the polymerisation and condensation of hydrocarbons and mineral oils by means of the silent-electric discharge and by alpha particles. The authors show that cathode rays produce the same results, and a method is described which is suitable for exact comparisons of various oils. Large quantities of gases, with hydrogen predominating, are obtained from the condensation of saturated oils, and only small quantities with unsaturated oils. Paper subjected to the action of cathode rays was found to give moisture, hydrogen, and carbon dioxide as the principal volatile products. Finally the importance of the reaction of oils and paper under the influence of the silent electric discharge is discussed in connection with the deterioration of impregnated paper insulated cable during use.

H. E. T.

429. The Less Volatile Oils of Salt Creek Crude. H. M. Smith. U.S. Bureau of Mines. *Technical Paper* 428.

In the above communication laboratory methods for the preparation of lubricating stock and its subsequent fractionation are described. The methods consist in (*a*) the topping of the crude oil at an elevated temperature by natural gas, (*b*) the separation of the solids and the liquids contained in the crude oil residuum by means of acetone, (*c*) the fractionation of the lubricating stock by steam distillation, (*d*) the separation of the steam cuts into two portions, one relatively insoluble and the other extremely soluble in acetone, (*e*) the vacuum distillation of the residua from the acetone extractions. The methods are applied to Salt Creek (Wyo.) crude oil and the viscosity, specific gravity, refractive index, molecular weight and ultimate analysis of the fractions obtained are given in the original. The data indicate the presence of two distinct groups of oil: One group relatively insoluble in acetone has a low viscosity-gravity ratio, low carbon-hydrogen ratio and relatively low unsaturation, and members of it are of the same type as the Pennsylvania light lubricants. Their composition indicates that they are members of series having the general formulæ C_nH_{2n-2} or C_nH_{2n-4}. The other group is extremely soluble in acetone and has high viscosity-gravity ratios, high carbon-hydrogen ratios and considerable apparent unsaturation. The hydrocarbons comprising it appear to belong to such series as C_nH_{2n-6} to C_nH_{2n-12}. Physical properties such as viscosity, specific gravity, index of refraction have been found to vary uniformly with apparent changes in constitution such as increased unsaturation.

N

The results now described are comparable with those obtained by Mabery from Cabin Creek, Teapot Dome and Sour Lake lubricating stocks and also with those of Wilson and Alibone from Burmah and Hardstoft crudes.

R. W. L. C.

Chemistry of Petroleum.

430. Sulphur Compounds in Pressure-cracked Naphtha and Cracked Naphtha Sludge. D. S. McKettrick. *Ind. Eng. Chem.*, Vol. 21, **6**, 585-592.

The naphtha used in this work was obtained by cracking a Californian Midway crude oil of specific gravity 0·953 at 15°C. It had an initial boiling point of 30°C. and an end point of 250°C., while its specific gravity was 0·786 at 15°C. and sulphur content 1·0 per cent. Two hundred and twelve litres of the cracked naphtha were extracted with liquid sulphur dioxide, six successive 25 per cent. by volume washes being employed. The extracted oil was submitted to a series of fractional extractions, first with aniline and then with ethylene glycol diacetate, at temperatures ranging from – 10°C. to – 20°C. The final yield was 13·6 litres of oil containing 5·5 per cent. of sulphur and having a specific gravity of 0·9 at 15°C. This extract contained 40 per cent. of all the sulphur present in the original naphtha. Finally, the sulphur compounds in the extract were further concentrated by fractional distillation. The distillation indicated the presence of theophene isomers, and these were identified by means of their reaction with mercuric chloride, which yielded mercuric chloride derivatives which were analysed. A second derivative was also formed and its composition determined as a confirmation. By this means the following compounds were identified, α-methyl-thiophene, β-methyl-thiophene, 2·3 dimethyl-thiophene, 2·0 ethyl-thiophene, 3·4 dimethyl-thiophene, 3·0 ethyl-thiophene and thiophene.

It has been further found that the sulphur compounds in the light oil, obtained by diluting and distilling cracked naphtha and sludge, are of a very different character, being sulphides of unknown structure and in all probability cyclic.

H. E. T.

431. Air Oxidation of Hydrocarbons Catalysed by Nitrogen Oxides. C. H. Bibb and H. J. Lucas. *Ind. Eng. Chem.*, 1929, **21**, 633-638.

I.—Natural Gas.

A summary of previous work on the subject of the oxidation of methane is given. The present work was undertaken largely with the idea of ascertaining optimum conditions for the production of formaldehyde.

In the presence of nitric acid vapour or its decomposition products, and at temperatures between 600 and 750°C., mixtures of natural gas and air give yields of formaldehyde up to 4 per cent. calculated on the total carbon present. The low concentration of nitric acid obtained by passing the gases through ordinary conc. nitric acid at 22°–24°C. suffices to catalyse the oxidation, and is, in fact, the optimum concentration (about 0·016 gm. nitric acid vapour per litre of gas mixture).

The presence of methanol in the oxidation products indicates that oxidation proceeds according to Bone's hydroxylation theory. Preliminary experiments showed a large number of solid catalysts to have little effect on the oxidation of methane to formaldehyde, probably owing to the preferential adsorption of formaldehyde resulting in further oxidation.

Experiments were made both on a small scale and on a larger scale. In each case the two streams—one of air, the other of natural gas—were measured by flowmeters, passed through wash bottles containing 66—67 per cent. nitric acid, then united and passed through the combustion tube, the central portion of which was heated in an electric furnace. The temperature of the furnace was measured by a thermocouple and regulated by a rheostat. Products from the small-scale plant were condensed in a series of U-tubes immersed in ice water, each tube having a water seal at the bottom. On the large-scale plant products pass through two water-cooled condensers, then a scrubbing tower for recovering all the formaldehyde.

The natural gas consisted of 85–87 per cent. methane, 3–7 per cent. ethane, nitrogen, carbon dioxide, and higher hydrocarbons. In the small-scale work the air-natural gas ratio was varied between the limits 2·5 to 1 and 7 to 1. The time of heating is an important factor. At the lower temperatures (450–600°C.) the gases must remain longer in the reaction zone in order to give a good yield of formaldehyde, but under these conditions some of the desired product decomposes, as shown by the relatively large amount of carbon monoxide in the effluent gas. At the higher temperatures (700–730°C.) the gases should remain not longer than 0·1 to 0·2 second in the reaction zone.

On the large-scale operations attention was given to the conditions affecting concentration of formaldehyde in the condensate obtained directly from the gas stream on cooling. Although the best yield on the basis of total carbon present was obtained with an air-natural gas ratio of 4 or 5 to 1, the concentration of formaldehyde in the condensate was lower than with a less efficient mixture, say, 1 to 1. In the latter case a maximum concentration of 25 per cent. was obtained when the temperature was 750°C. and the gases remained in the heated zone for 0·13 second. A 50 per cent. increase in the reaction time under similar conditions reduced the concentration to 22 per cent., and a 150 per cent. increase in the time gave a still lower concentration and lower yield. On the other hand, a 4 to 1 air-natural gas ratio was made to give a 50 per cent. increase in formaldehyde yield over the first case cited, yet the concentration of formaldehyde in the condensate was halved.

These dilute solutions of formaldehyde can be concentrated to commercial strength by fractional distillation.

II.—Benzene.

Work on similar lines with a view to obtaining a maximum yield of phenol is described. The previous maximum yield of phenol by air oxidation of benzene over solid catalysts was 0·3 per cent., whereas 5 to 5·5 per cent. yields have been obtained in the present work. These yields again bear out Bone's hydroxylation theory.

The catalytic effect of the oxides of nitrogen in the gas-phase oxidation of benzene may be due (*a*) to an increase in the concentration of active (atomic) oxygen, or (*b*) to the greater oxidising power of nitrogen peroxide. Continued oxidation of phenol would presumably produce dihydroxybenzenes, quinone, and finally maleïc acid. The latter can be produced with solid catalysts which selectively adsorb phenol and hold it in the reaction zone Hence a gaseous catalyst is again valuable, as the phenol can be swept out of the heated zone as soon as formed.

Work was again carried out on two scales. Each plant comprised two flowmeters, admitting air in the one case to nitric acid wash bottles, in the other case to weighed benzene wash bottles maintained at about 70°C. The two streams were combined, led through a reaction tube in the electric

furnace, and the products cooled. In the small-scale apparatus a silent electrical discharge was employed to precipitate the fume, after which the products passed through U-tubes immersed in ice water. On the large-scale plant the cloud was satisfactorily settled in large settling jars. The products then passed through four large wash bottles containing light lubricating oil, which quantitatively recovered benzene from the tail gases.

Phenol was extracted from part or all of the condensates, and from part of the oil with dilute caustic soda, acidified, diluted, and estimated as tribromophenol. In experiments on the small-scale plant the nitric acid was at room temperature; on the large scale it was maintained at about 40°C.

A maximum conversion of about 5·5 per cent. of the benzene was possible under different conditions, but in the first series of results (small-scale plant) a fair yield of phenol was always accompanied by excessive oxidation, judging by the dark colour of the condensates and the formation of carbon dioxide and formaldehyde. Optimum conditions in this series seem to be: Temperature 550–700°C., air-benzene ratio between 1·5 to 1 and 2·5 to 1, heating time 1 to 2 seconds. The use of 75 per cent. and 95 per cent. HNO_3 produced black tarry condensates.

In the second series (large scale) experiments the excessive oxidation was largely eliminated by running the gas through the furnace in 0·2 to 0·5 second, and the yield of phenol was expressed, firstly, as a percentage of the total benzene; secondly, as a percentage of the benzene which was attacked. The best yield in this series was 52 per cent. on the benzene attacked, or 3·5 per cent. of the total benzene, the furnace temperature being 700°C., and nitric acid temperature 40°C. Six mols of phenol were produced per mol of acid vaporised (the maximum in any experiment was 9·7 mols phenol per mol acid). Concentration of nitric acid in the vapour phase was important. When 50 per cent. and 60 per cent. acids replaced the 67 per cent. acid only a trace of phenol was produced, whilst increasing the temperature of the concentrated nitric acid up to 80°C. produced maximum yields of phenol on the *total* benzene, although excessive oxidation took place at the same time, and only 27 per cent. of the benzene attacked was converted into phenol. L. H. A.

432. Hydrogenating and Treating Carbonaceous Materials. M. Hofsäss. U.S.P. 1,711,499, May 7, 1929. Appl. Nov. 30, 1926; Ger., Dec. 2, 1925.

In the method of manufacturing benzene from carbonaceous products by the Bergius process considerably increased yields can be obtained and higher temperatures used without coking if the carbonaceous matter is heated under pressure of hydrogen in the presence of a hydride of anthracene and its bromologues, as well as inorganic catalysts, to a temperature at which the hydrogen in the hydride is rendered unstable. The product so produced is fractionated and a fraction therefrom heated to a higher temperature to separate the new material of the organic hydride. H. S. G.

433. The Thermal Decomposition of Methane. Part I. Decomposition in Silica Bulbs. G. C. Holliday and H. C. Exell. *Jour. Chem. Soc.*, May, 1929, pp. 1066-1074.

In this paper the authors describe the results of static experiments on the decomposition of methane in silica and porcelain bulbs over the temperature range 800–1,200°C. In all cases there is a very rapid initial decomposition, followed by extremely slow dissociation, while the percentages of methane remaining after a period of one hour are much greater than

would be expected from true equilibrium values. The effect of varying the pressure showed that the dissociation increased as the pressure decreased. If hydrogen is added initially to the methane, the rate of reaction is exactly the same as if dissociation of pure methane had already reached a point corresponding to the composition of the initial mixture. If more than the equilibrium quantity of hydrogen is present the decomposition is extremely slow. Nitrogen and carbon monoxide had no effect on the rate of reaction or final amount of dissociation, if the initial partial pressure of the methane remained the same. At a constant total pressure, however, they act as diluents, thus increasing the percentage decomposition. Ethane was found to decompose to the same equilibrium mixture of hydrogen and methane as methane itself, while by using porcelain for the reaction bulbs similar results were obtained. Results obtained over a wide range of pressures show that the values of P_{CH_4}/P_{H_2} are constant, the effect of temperature on this constant being exactly analogous to its effect on true equilibrium. The retardation of the velocity of reaction is so marked in all cases that for practical purposes a false equilibrium is obtained.

By coating one of the reaction bulbs on its inner surface with nickel, the velocity of decomposition at 900 deg. was enormously increased, no retardation taking place. Since only 3 per cent. of methane remained at the end of 18 minutes, the indication was that a state of true equilibrium was being rapidly approached.

The results obtained by the authors show that the decomposition of methane is not a true unimolecular reaction, while the apparent equilibrium observed may be due to the following causes:

(1) The solid deposited by methane may be a hydrocarbon, having a greater vapour pressure than carbon.

(2) The reaction is being strongly retarded by selective absorption.

H. E. T.

434. Production of Homogeneous Mixtures of Aliphatic Mineral Oils or Distillates and Aliphatic Alcohols. J. Kirschner. E.P. 276,357. Dec. 10, 1928.

Homogeneous mixtures (emulsions) of liquid aliphatic mineral oils or distillates and water containing aliphatic alcohols are prepared by subjecting mechanically prepared mixtures of the components at raised temperatures to the action of nascent hydrogen. Lower boiling fractions of petroleum, *e.g.*, benzine, kerosine, gas oil, are particularly suitable for the process, but heavy fractions or even crude petroleum may be used, ethyl or methyl alcohol may be employed. The process may be carried out by causing the mixtures with the addition of organic acids or salts (*e.g.*, oxalic acid or oxalates) to pass over or through heated metals or suitable alloys of m.p. above 100°, *e.g.*, lead-nickel alloys. Or the mixtures with the addition of volatile inorganic acids such as hydrochloric acid may be caused to pass in vapour form over granulated metal such as zinc. W. S. N.

435. Manufacture and Production of Mono and Polyhydric Alcohols. J. Y. Johnson and I. G. Farbenindustrie Aktiengesellschaft. E.P. 309,200. Mar. 28, 1929.

Monohydric and/or polyhydric alcohols are produced by subjecting a crude reaction mixture of the condensation of an aldehyde or a ketone with the same or other aldehydes or ketones to a catalytic hydrogenation at atmospheric or elevated pressures (20—30 atm.), and if desired at an elevated temperature (50°), the uncondensed aldehydes or ketones being removed.

if desired, prior to hydrogenation, but without separating the condensation product from the condensing agent, the latter being neutralised if desired. The hydrogenation may be performed in the presence of diluents (water, or primary aliphatic alcohols). The catalysts may consist, generally, of metals of the 1st or 8th groups of the periodic system (in particular, nickel) or of mixtures of the same with each other or with activators such as oxides, hydroxides, silicates or phosphates of the same or other elements, preferably in conjunction with a carrier such as kieselguhr or fuller's earth.

Thus, 20 parts of crude aldol containing about 30 per cent. of acetaldehyde (obtained, *e.g.*, by condensing acetaldehyde with 2N- caustic potash solution) are diluted with 10 parts of alcohol and treated with hydrogen at about 50° and 20—30 atm., in a stirrer autoclave containing 1 part of a 20 per cent. nickel catalyst on fuller's earth. The temperature is maintained at the specified level by cooling. When, after 8—10 hours, the absorption of hydrogen ceases, the temperature is raised to about 80°, and stirring continued until the pressure ceases to drop. After separating the catalyst by filtration the solvent is distilled off together with the ethyl alcohol derived from the residual acetaldehyde, and the 1.3-butylene glycol fractionated in vacuo. The yield corresponds theoretically with the aldol present in the crude mixture and amounts to about 70 per cent. of the aldehyde used, the remaining 30 per cent. being recovered as alcohol.

Aldol (10 parts) is diluted with water (5 parts) and hydrogenated at 80°—100° using a nickel catalyst; after separating the catalyst the reaction product, which is in two layers, gives a good yield of primary *n*-butyl alcohol together with small quantities of 1.3-butylene glycol and alcohol, derived from the unreacted aldehyde. The greater part of the aldehyde may be recovered from the crude aldol prior to hydrogenation by neutralising and distilling in vacuo.

The crude aldol condensation product from *n*-butyraldehyde and dilute caustic soda is neutralised with acetic acid and the excess of aldehyde removed in vacuo; half its volume of alcohol is then added and hydrogenation carried out at 70—80° and 20—30 atm., using 1 per cent. of nickel catalyst (calc. on the aldol present) on fuller's earth. By fractionation of the solution after removing the catalyst an excellent yield is obtained of α-ethylhexanol, b.p. 180—181°.

Aldol (660 parts) is added to 30 per cent. formaldehyde (728 parts) and N-caustic soda solution (100 parts by volume) slowly run in at 15—20°. The pH value of the charge is then adjusted to 8—10. When, after about 12 hours, the aldehyde content of the mixture has diminished to one-half, the crude condensation product is neutralised with acetic acid, diluted with half its volume of alcohol, and hydrogenated at 60° and 20—30 atm., using 1 per cent. of nickel on fuller's earth. Water is removed from the filtered reaction product by distillation in vacuo, leaving a clear, syrupy, water-soluble product that can be employed after removal of the deposited sodium acetate as a plasticising, gelatinising or softening agent.

Similarly a pale syrup, applicable to numerous purposes, is obtained from the mixture of sugars produced by condensing formaldehyde with a dilute solution of lime, by filtering and concentrating, diluting with alcohol and hydrogenating at a temperature range gradually rising to 100°, and at about 25 atm., using a nickel catalyst on kieselguhr activated with chromium.

Formaldehyde solution (1,000 parts of 4 per cent.) is heated for about 45 minutes at 130° in a closed vessel with 2.5 parts of sodium carbonate, and the reaction product directly reduced with hydrogen at 100° and 25 atm. in the presence of a nickel catalyst (with 40 per cent. of nickel) on fuller's earth. Filtration and concentration yields a highly viscous liquid

from which a considerable yield of glycol and glycerol, in addition to higher polyhydric alcohols, can be obtained by fractional distillation.

In the reduction of aldol after neutralising and removing the excess of aldehyde, a catalyst may be employed consisting of 40 parts of cobalt on 60 parts of kieselguhr; or the catalyst may consist of 27 per cent. of copper, 8 per cent. of nickel, 15 per cent. of chromium (as chromous oxide) and 50 per cent. of kieselguhr. W. S. N.

436. Recovery of Oxidation Products of Solid Hydrocarbons, Waxes and the Like. J. Y. Johnson and I. G. Farbenindustrie Aktiengesellschaft. E.P. 309,875. Apr. 8, 1929.

It is proposed to recover the products of incomplete oxidation of solid hydrocarbons, *e.g.*, wax, by filter pressing at a temperature below the m.p. of the original material. If desired, the acid portion of the crude oxidation product may be converted into salts, which may then be removed and the remaining material cooled and pressed.

Thus, a crude oxidised soft paraffin wax (100 parts), containing 50 per cent. of acids, is hydraulically pressed at 15° under a pressure of about 50 atm. The recovered solid (about 30 per cent.) consists to the extent of 95 per cent. of paraffin wax. The liquid portion (about 70 per cent.) is a pale oil, which does not deposit further wax on prolonged standing and cooling, and which may be used for the preparation of soaps or emulsifying agents or in the textile industry for oiling or smoothing or as material for the production of sulphonated oils. The same oxidation product is treated with a slight excess of aqueous alkali and the non-acid constituents which separate from the soap solution are removed and pressed as before. A solid residue, about 50 per cent., mainly consisting of paraffin wax, is left, whilst 50 per cent. of liquid constituents drain off, consisting chiefly of alcohols of high mol. wt. W. S. N.

437. Oxidation of Aliphatic Compounds of High Molecular Weight. J. Y. Johnson and I. G. Farbenindustrie Aktiengesellschaft. E.P. 310,069. Apr. 17, 1929.

It is already known to oxidise paraffins by means of air or oxygen, at a pressure of at least several atm. in the presence of caustic alkali or alkaline earth in such a quantity that the main portion of the fatty acids formed is instantly converted into soaps, which act as accelerators in the oxidation of the paraffin.

It is now found that excellent yields of very light coloured and highly oxidised products are obtained from aliphatic compounds of high mol. wt. (especially paraffin hydrocarbons, *e.g.*, wax) by heating in the presence of a gaseous oxidising agent (air, oxygen or gases containing the same, nitrogen oxides or mixtures of these agents), if the treatment be performed with the aid of a catalyst consisting of a salt of an organic acid with an alkaline earth metal or magnesium or aluminium or a mixture of such salts, but in the absence of free alkalies or alkaline earths. Compounds that will dissolve in the hydrocarbon under treatment or in the products are suitable, *e.g.*, stearates, abietates, anthranilates or cinnamates of the metals mentioned. The catalysts are advantageously employed in conjunction with inert fillers (aluminium or ceramic materials). Temperatures of 80—180° are employed.

Thus air is blown for several hours through 1,000 parts of soft paraffin wax, in which 1 part of barium stearate has been dissolved in the warm, in a tower filled with aluminium rings, at 150°. An oxidation product is obtained, only slightly tinged with yellow, of the consistency of ointment and having acid value 86 and sap. value 236. A distillate is also obtained

consisting of readily volatile oxidation products. A white product of the consistency of ointment, having acid value 103 and sap. value 130, is obtained by air-blowing at 130° soft wax, in which 0.1 per cent. of barium or calcium cinnamate has been dissolved. W. S. N.

438. Purification of Synthetic Butyl Alcohol. J. Y. Johnson. E.P. 312,512. May 30, 1929. (Assr. to Farbendind. A.-G.). Appl. June 20, 1928.

The process consists in treating synthetic butyl alcohol prepared by the catalytic hydrogenation of crotonic or butyric aldehyde with chlorine, bromine, hypochlorous or hypobromous acid, and separating the butyl alcohol from the halogen derivatives so formed. The method of preparation gives a material free from nitrogenous compounds. W. H. T.

439. Characteristics of Non-Explosive Oxidation of Propane and Butanes. R. N. Pease. *J. Amer. Chem. Soc.*, 1929, **51**, 1839–1856.

The paper deals with the reactions of the hydrocarbons when passed through a heated tube with varying proportions of oxygen.

The gases were led through flerometers at specific rates, and the mixture was passed through a pyrex glass reaction tube in an electrically-heated furnace, maintained at a definite temperature until equilibrium was reached, when the products were passed to a condensing trap, and the gaseous products analysed for CO_2, unsaturates, O_2, CO, H_2, CH_4, and original hydrocarbon. The liquid products were not examined.

Reactions were classified into three types:—

(I.) Unsaturates by dissociation:

$$C_3H_8 \rightarrow C_3H_6+H_2$$
$$C_3H_8 \rightarrow C_2H_4+CH_4$$

estimated by volume of CH_4 plus H_2.

(II.) Unsaturates by oxidation:

$$C_3H_8+\tfrac{1}{2}O_2 \rightarrow C_3H_6+H_2O$$
$$C_3H_8+\tfrac{1}{2}O_2 \rightarrow C_2H_4+CH_2OH, \text{ etc.}$$

estimated by volume of unsaturates less volume of CH_4 less volume of H_2.

(III.) Aldehyde or carbon monoxide reaction:—

$$C_3H_8+O_2 \rightarrow C_2H_5{\cdot}CHO+H_2O$$
$$C_2H_5{\cdot}CHO+O_2 \rightarrow CH_3{\cdot}CHO+H_2O+CO.$$

estimated by volume of hydrocarbon consumed less volume of unsaturates.

Propane.—Reactions I. and II. always appeared together, sometimes one and sometimes the other predominating. Unless reaction III. was taking place reactions I. and II. did not appear until the temperature reached 550—600° C., *i.e.*, conditions conducive to thermal dissociation had to be approached before any reaction occurred. Reaction II. was found to be contingent on the possibility of dissociation, and at the same time promoted dissociation. These two reactions gave evidence of being normal homogeneous gas reactions, first order with respect to propane, but subject to excitation by reaction III. in amount which was small at the lower temperatures, but increased as the temperature was raised.

Reaction III. was wholly different; it was liable to commence at 300—350° C., was promoted by excess of hydrocarbon, but depressed by excess of oxygen, or dilution with other gases. Experiments with O_2, CH_4, N_2, CO_2,

and H_2 as diluents showed marked suppression, the amount increasing in the order given, and increasing with rise of temperature, showing that under these conditions the reaction had a negative temperature coefficient. The reaction was likewise suppressed by broken glass packing, owing to the more ready deactivation of excited new molecules; but the glass exerted a positive catalytic effect, shown by further suppression after poisoning with potassium chloride. Reduction of heating time by the use of a small reaction tube, and reduction in pressure to a few mms. of mercury had similar effects in suppressing the reaction. The author considered these facts to point conclusively to a chain reaction as postulated by Edgar and Pope for the higher hydrocarbons.

Butanes.—Reaction was in general more ready than with propane, *n*-butane being the more reactive of the two. Suppression of the reaction was in general more difficult. The "free" reaction in the empty tube passed more readily into explosion. With the smaller reaction tube the system of interpretation appeared to be strained by the appearance of excessive quantities of unsaturates; dissociation and CO formation were greatly decreased.

The author concluded that there was no essential difference between the reactions of the butanes with oxygen and those of propane. The aldehyde with one less C atom than the hydrocarbon should be formed in a fairly pure state by oxidation at the lowest possible furnace temperature with excess of hydrocarbon. High temperatures and maximum suppression were thought to provide a good method of preparing olefines. It was thought that any catalyst would play a dual role in that it would suppress reaction III. apart from its specific action on the other reactions. L. H. A.

440. Mechanism of the Vapour Phase Oxidation of Isomeric Octanes. I. Normal Octane. J. C. Pope, F. J. Dykstra and Graham Edgar. *J. Amer. Chem. Soc.*, 1929, **51**, 1875–1889.

The subject is of interest in throwing light on the mechanism of the oxidation of commercial motor spirits. Mixtures of octane vapour and air were passed through an electrically heated pyrex glass tube, provided with thermocouples and arrangements for sampling the gas at any desired point. Octane was fed from burettes to a preheater, vaporised in a stream of nitrogen, and passed to a large annular tube projecting into the furnace, surrounding an inner annular tube through which the requisite amount of oxygen was passed. The two streams were allowed to mix at a point where furnace temperature had been attained. The oxygen and nitrogen streams were maintained at a constant rate, in the proportions of air, while octane was fed at the theoretical rate for complete combustion. The products of oxidation were passed through a condensing system after which the gases could again be sampled. Gas analyses were made for CO_2, O_2, and CO; H_2 and CH_4 were never detected. Acid, aldehyde, peroxide, and water were estimated in certain condensates.

Experiments were carried out in two ways: (*a*) At constant furnace temperature; (*b*) with slowly rising temperature. Constant temperature runs showed the reaction to be largely localised in the first 20 per cent. of the tube length, both from the temperature gradient and gas analyses at various points. Liquid products invariably contained aldehyde and water.

Runs with slowly rising temperature were the most informative. Reactions may be divided into three temperature zones: 200–270° C., 270–650° C., and above 650° C. Between 200–270° C. the O_2 consumption rises from nil to 2 mols per mol of octane, and CO_2 formation rises from zero to 0·4 mol. At 270° C. CO formation commences and rises rapidly to 0·5 mol. A feeble

white glow fills the tube intermittently, moving counter current to the gases, and accompanied by pulsations in the flowmeters and a more rapid rate of rise of reaction temperature. The white glow finally settles at the gas inlet. The O_2 consumption rises rapidly to about 4·7 mols at 350° C., after which the curve flattens, rising to 5·5 mols at 550° C., then remaining nearly constant to 650° C. CO rises to 2 mols at 350° C., after which it increases steadily to 3·5 mols at 650° C.; CO_2 rises to .1 mol at 400° C., thereafter remaining constant to 650° C. Above 650° C. the reaction becomes almost explosive: pulsations set in again accompanied by bright blue flashes moving with the gas stream. O_2 consumption and CO_2 formation rise rapidly, CO falling to a very low figure. The luminescence of the second stage persists in the intervals between flashes.

Heptaldehyde and butyraldehyde were also studied and found to have somewhat similar characteristics: heptaldehyde oxidised more readily, and butyraldehyde less readily, than octane.

The data indicate that below 650° C. the primary oxidation product of octane is octaldehyde, and that this aldehyde is further oxidised to aldehydes of lower molecular weight:—

$$CH_3(CH_2)_5CH_2{\cdot}CH_3+O_2=CH_3(CH_2)_5CH_2{\cdot}CHO+H_2O \quad . \ . \ . \ (1)$$

Main reaction, $CH_3(CH_2)_5CH_2{\cdot}CHO+O_2=$
$$CH_3(CH_2)_5CHO+CO+H_2O \quad . \ . \ . \ (2A)$$

Side reaction $CH_3(CH_2)_5CH_2{\cdot}CHO+1{\cdot}5O_2=$
$$CH_3(CH_2)_5{\cdot}CHO+CO_2+H_2O \quad . \ . \ . \ (2B).$$

Reaction (2) is assumed to continue in the same way yielding each time an aldehyde with one less C atom. This hypothesis is borne out as follows. The oxygen consumption of butyraldehyde and heptaldehyde is shown to be identical with that calculated from $CO+1{\cdot}5CO_2$ while the oxygen consumption of octane exceeds this figure in all cases by unity (Reaction 1). Any attack of the aldehyde chain other than at the end would involve more oxygen consumption for a given quantity of carbon oxides.

The CO_2 reaction is unaffected by the rate of heating and is only a small factor at temperatures below 650° C. The CO reaction appears to be entirely different. Its sudden appearance at 270° C. and its accompaniment by pressure surges and luminescence suggests it to be a photochemical "chain" reaction. The surges and luminescence at the start of the second stage are presumed to correspond with the sudden reaction initiated by accumulated activated aldehyde molecules.

The flattening of the O_2 consumption curve with increase of temperature may be explained by the progressive formation of lower and more difficulty oxidised aldehydes. L. H. A.

441. Condensation Products from Crude Cresol and Aliphatic Ketones. Schering-Kahlbaum Aktiengesellschaft. E.P. 179,856. Jan. 25, 1929.

It is already known to manufacture condensation products from aliphatic ketones and *m*-cresol or *p*-cresol (cf., E.P. 273,684; 273,685; 276,010; 279,855). It is now proposed to use crude cresol, consisting of *m*- and *p*-cresol, for the purpose. Thus, a mixture of crude cresol (4 parts) and acetone (1 part) is saturated with gaseous hydrogen chloride, allowed to stand 2 hr. at 40—80°, and the excess of cresol removed by means of steam or in vacuo. The residue may then be distilled in vacuo and then yields a yellow, highly refracting mass that solidifies to a resin. When acetone is the ketonic component the two reaction products may be separated if desired by dissolving the mixture in ether. After a short time crystals separate which are the pure

m-cresol-acetone product containing ether of crystallisation and which have m.p. 73—75°; the *p*-cresol-acetone product remains in the mother liquor. When acetone is the ketonic component, the product may be worked up by hydrogenation into mixtures of thymols or menthols. W. S. N.

Crude Oil.

442. Oil Treating Process and Apparatus. J. R. Hall. U.S.P. 1,718,141. June 18, 1928. Appl. May 25, 1927.

The invention relates to an improved method and apparatus for separating water and bottom settlings and other foreign matter from oil, especially crude petroleum. The object is to effect a clean separation of oil and foreign matter continuously by leading the oil by combined automatic suction and a force pump to a centrifugal separator. The latter comprises a hollow shaft, a rotary drum having two independent lateral ducts side by side connecting the hollow axis with the periphery, and a port between the two at the periphery. An ejection valve is provided for the impurities of greater specific gravity, and is automatically controlled at a definite pressure. A lateral duct leads the clear oil centripetally to the shaft, thence to a storage tank, while an outer casing collects the extruded impurities and delivers them to floor level.
W. H. T.

443. Technical Methods of Breaking Crude Oil Emulsions. A. Lottermoser and N. Calantar. *Kolloid Zeitschrift.* Vol. 48, No. 2 (179–184).

The four chief groups of patented processes for the removal of water from crude oil emulsions are described. Numerous patents are given, but the following appear to be the most important :—

I.—Electrical Methods.

In 1919 W. G. & H. C. Eddy (*Jour. of Ind. Eng. Chem* 13, 1016, U.S.P. 1,554,528) introduced a method of dehydrating crude oil in which the crude oil emulsion was added continuously to a galvanised steel container and subjected while stirring to a potential difference of 11,000 volts by means of an alternating current. They state that the output of such an apparatus should be 300–1,000 barrels of oil per day. Three years later Eddy introduced another method (U.S.P. 1,565,992) dealing with the same type of emulsions in which the emulsions were stabilised by finely divided solid bodies. N. Bataafsch, Skaer, Girwin, Quinby and many others of the Standard Oil Co., San Francisco, utilised a substance which conducted electricity and enabled both phases of the emulsion to have an equal electrical charge.

II.—Mechanical Methods.

A. *Centrifuges and other Apparatus.*—A Sharples Super Centrifuge is used so as to attain a velocity of 40,000 r.p.m. W. A. White (E. P. 250,360, 1925) used a filter and a large surface to separate emulsions. N. Metta (Oesterr. P. 102,957) constructed a pressure apparatus which was partly filled with a molten metal.

B. *Heat Treatment.*—The Thermal Industrial & Chemical Research Co., Ltd. (B.P. 221,528) passed the initial substance through a tube lying in a metal bath whose temperature only exceeded the boiling point of the highest boiling distillate by a few degrees in order to avoid cracking. The Badische Anilin und Sodafabrik (G.P. 354,202) heated the crude in a closed vessel under excess pressure to 200°. The water was then blown from this vessel.

C. *Filters.*—Hatschek found in 1908 that clear water flowed through a $CaCO_3$ filter but oil was retained. (E.P. 26,228.) Vulcanised caoutchouc and iron sulphide allowed the oil to pass through and retained the water. J. McKenzie (*Oil & Gas. Jour.*, 1925, No. 2,758) filtered the emulsions through "Celit," a very fine Kieselguhr which was arranged in filter-presses. Such a press had a surface of 600 sq. ft., and on an average 1,500 gallons of oil were obtained when Californian crude oil was filtered.

III.—Various Addition Compounds.

A. *Adsorption Media.*—The addition of weakly alkaline substances effect a decomposition of the emulsion—*e.g.*, brown coal ash, coal ash, blood charcoal, wood charcoal, bone ash, Fe_2O_3, bleaching earths, fuller's earth, clay lime, Kieselguhr, burnt shale, $BaSO_4$ and kaolin. In the dehydration of shale-tar 200 kgms. of coal-ash were necessary to reduce the water content from 75 per cent. to 4.

B. *Addition of Electrolytes.*—R. Barry (U.S.P. 1,593,893) heated the oil with a mixture of 0·1–1 per cent. NaCl and pulverised clay, and allowed the oleaginous and aqueous phases to separate. Other patents recommend the application of iron chloride and sodium carbonate.

C. *Various Solvents.*—Two influences prevail regarding the stability of emulsions, the stability due to the presence of various high-molecular hydrocarbons—namely, petroleum resins, asphalts and asphaltenes; and on the other hand the salts dissolved in the occluded water react with the free organic acids of petroleum, forming salts of the alkaline earths, chiefly calcium soaps. The protective film must either be dissolved or a tendency to form emulsions of the opposite type must be developed, which is equivalent to neutralising the effect of the colloid already present. T. Kuczynski (G.P. 406,818) added benzine, benzol, alcohol, ether, and found that by finally increasing the temperature the solubility of the emulsion increased and then the emulsion broke. Gurwitsch maintains that in this process the surface tension reduces the effect of the added solvents which remove the asphaltic substances from the boundary surface oil-water. It was also found that definite mixtures of benzine, alcohol and ether have the property of precipitating in certain concentrations petroleum asphalts. J. Bregeat (French P. 608,154) washed the emulsions continuously with or without pressure with solutions of the phenol-cresol series, their hydrated derivatives or those of naphthol, aromatic sulphonated compounds or mixtures of these. Amongst other compounds added were ethers and sulphonates of fatty acids, and hydroxylated, halogenated, sulphonated and nitrated products, amines, ethers and their soaps.

D. *Addition of Colloids.*—E. Ayres (*Chem. Abstr.* 15, No. 22, 3904) mixed the emulsion with a small quantity of a substance which is soluble in petroleum but contained a protective colloid soluble in water. Starch is normally insoluble, but it can be combined with substances which render it soluble in oil. Barnickel describes in American patents the addition of solutions of highly colloidal coagulating soaps, mixtures of aqueous solutions of the oleate and resinate of sodium, various other oleates, lactones, naphthenic acids, resins, fatty acids, linseed oil, olive oil, aqueous glycerin solution, etc., and of dioxystearic acid and ammonium soaps, or alkali soaps of a polycyclic sulpho-acid. Matthew and Crosby added a 1 per cent. aqueous solution of Tret-o-lite to an emulsion in the relation of 0·1–1 per cent. by weight, and

after shaking the phases separated and then were precipitated. The concentration of the substances dissolved in Tret-o-lite was varied according to the property of the emulsions to be treated until the optimum relation was attained.

IV.—By-Products

A. Calantar utilised as a demulsifying agent the distillation product obtained from the portion insoluble in water left from the spent lye after sulphuric acid refining. Nugent (E.P. 177,498, 1920) found that an emulsion of 50 per cent. benzol in water containing 0·4 per cent. gelatine remained stable on centrifuging at a speed of 2,000 r.p.m., but the constituents separated when the wheel was arranged so as to revolve at the rate of 3 r.p.m. A separation into the components was dependent on the size of the air space in the tube. Shepphard, Beckhold, Dede and Reiner established an optimum time of 10 minutes slow shaking for the demulsification of emulsions with finely divided solid bodies. Conradson determined the emulsifying capacity of an oil (*Journ. Ind. Eng. Chem.* 9, 166–167) by passing steam from a steam-generator for quarter-hour into a calibrated vessel containing 20 ccms. water and 100 ccms. illuminating oil. The vessel was then placed for one hour in water at 54·5° C., and the number of ccms. of water emulsion and oil noted.

In conclusion, there appears to be no universal method for breaking crude oil emulsions, as many media give good results for one type of emulsion but are unsuitable for others. W. S. C.

444. Treating Emulsions. R. Van A. Mills. *Oil and Gas Journ.* April 15, 1929, pp. 45, 150.

A unique method has been devised for treating emulsions formed from high gravity crude oil. The method depends primarily upon utilising the salt water accompanying the oil as a dehydrating agent, and also upon using the natural heat in the deep well fluids to accelerate the dehydration. After passing through gas separators, the oil, salt water and emulsion, enter the top of a separating tank, and flow downwards through a flume to the bottom of the tank. The tank contains warm salt water at a temperature of approximately 90° F. The fluid discharged from the bottom of the flume floats upwards in non-turbulent motion, during which process the emulsion is broken down, while the clean oil containing less than 0·5 per cent. of emulsion separates out at the top of the tank from which it is periodically withdrawn to storage. H. E. T.

445. Distillation of Oil. A. E. Miller. Assr. to Sinclair Refining Co. U.S.P. 1,714,097. May 21, 1929. Appl. June 11, 1927.

Crude petroleum oils are stripped of their lighter constituents and then maintained at a temperature between 575° and 650° F. in contact with a body of fuller's earth substantially co-extensive therewith but supported free from the bottom of the still. The evolved vapours are subjected to refluxing action, and the reflux condensate, together with fresh make-up oil, stripped of all light fractions before being returned to the still. H. S. G.

446. The "Hot Water" Process of Breaking Down Emulsions. T. F. Smiley. *Oil and Gas J.* 1929, 27, 48, 30.

The emulsion problem has been met with satisfactory results by the adaption of the domestic water heater. An ordinary oil-field boiler can be converted into equipment for breaking down emulsions by introducing a pipe coil in the firebox where it will be in immediate contact with the gas flame.

The emulsion as it comes from the well, flows through the coil which is kept heated to temperatures ranging from 120–140° F. A thermostat automatically prevents the temperature from reaching a higher degree than 140.

W. H. T.

447. Apparatus for Chemically Treating Natural Oils. J. L. Taylor and C. L. Howser. U.S.P. 1,717,744. June 18, 1929. Appl. Oct. 26, 1926.

The primary object of the invention is to provide an apparatus constructed and arranged in such a manner that crude oil can be conducted directly from a well to be mixed with a chemical treating solution. The mixture of oil and solution is subjected to heat in order that the basic sediment in the crude oil can be removed therefrom after the mixture has been conveyed to a settling tank. The apparatus is so constructed as to be continuous in action and capable of regulating the quantities of treating solution in order to obtain the best results. Air under pressure or natural gas can be used separately or together to insure proper feeding of the chemical treating solution to its point for thorough mixing with the crude oil.

W. H. T.

448. Silica Gel as a Reagent for the Extraction of High-Sulphur Oils from Crude Petroleum. (Preliminary Paper.) Gladys E. Woodward. *Ind. Eng. Chem.*, 1929, **21**, 693–695.

The paper indicates the possibility of making an effective separation of sulphur compounds in crude petroleum by a series of fractional adsorptions with silica gel followed by fractional removal of the adsorbed material by means of organic solvents which have different displacing powers toward the adsorbed material.

By washing the silica gel, charged with sulphur compounds, first with the least effective solvent, and then with the more effective ones in order different fractions of oil were obtained. The solvents and their order of use were: naphtha, benzene and ether. These removed about 90 per cent. of the adsorbed material, and the remainder was obtained by dissolving the gel in 10 per cent. caustic soda at 30° C., and extracting with ether, which left a trace of fine black material soluble in benzene.

In adsorbing the sulphur compounds in silica gel filtration of a crude oil through the gel was found to be too slow, hence a sulphur-free naphtha was used as solvent, and the whole mechanically stirred with silica gel. In the case of Inglewood crude petroleum commercial silica gel effected an 18 per cent reduction in sulphur content (from 2·3 to 1·9 per cent.) when agitated with the naphtha solution, and the extracted adsorbed oil in every case had a higher sulphur content than the original oil, the highest content occurring in the oil removed by benzene. Holmes silica gel (which is more porous) had twice the desulphurizing action, and the extracted materials contained nearly twice as much sulphur as those from the ordinary gel. Most of the coloured constituents in the oil were also removed by Holmes gel.

It was found possible to effect further concentration of the sulphur compounds by giving the extracted material another treatment with silica gel. Tables are given showing results obtained from Inglewood crude petroleum.

L. H. A.

449. Crude Oil Flows Downward into Well. *Oil and Gas J.*, 1929, **27** (50), 31, 148.

An Oil Flow Indicator invented by E. L. Trout shows that oil flows downward from a higher point structurally into a well, and it is claimed that by following the indicated flow of the oil not more than 2 to 5 per cent. of wells drilled

should be dry. The apparatus consists of a compass and a vane, the latter being enclosed in a perforated barrel through which oil, gas or water can flow. The compass is in a sealed box below the indicator. When in the desired position, the vane and compass needle are released and immediately the instrument is raised they are locked in position. It is stated that the indicator determines the direction of maximum flow and enables the productive horizon to be followed with a minimum of dry holes. In re-pressuring operations it enables the natural forces to be aided rather than combated. G. S.

Motor Spirit.

450. Process for Refining Hydrocarbons. J. C. Black and J. W. Weir. U.S.P. 1,709,518, April 16, 1929. Appl. March 2, 1923.

A process is provided for refining hydrocarbons, whereby certain distillates are fractionated into a merchantable gasoline without redistillation. The process consists in vaporising the hydrocarbons, and passing the vapours to the lower part of a fractionating tower. As the vapours rise up the tower part are condensed, and these are withdrawn from the bottom of the tower. The withdrawn condensate is circulated back to the upper part of the tower, and allowed to flow back down the tower, thus bringing it in intimate contact with the mixed vapours ascending the tower. By this means vapours having the same composition as the circulated hydrocarbons are separated from the ascending vapours. Part of the separated hydrocarbons which is in excess of that required for circulation through the tower are returned to the still. Some of the condensed hydrocarbons are subjected to a further fractionation in a second apparatus, operated at a different temperature. The residual vapours from both towers are separated and condensed.

H. E. T.

451. Manufacture of Motor Fuels. H. D. Elkington. E.P. 312,245, May 17, 1929. Appl. Jan. 17, 1928.

The invention has for its object the production of a motor fuel which contains dispersed therein a solution or dispersion in a suitable medium of any anti-knock dope whether soluble in the original fuel or not. The following example illustrates the method of carrying out the invention: An aqueous solution of potassium gallate is dispersed in benzine so as to produce a dispersion containing 1 gm. of potassium per litre of fuel. W. H. T.

452. Continuous Process for the Refining of Mineral Oils. E. T. Hesslé. U.S.P. 1,661,826, March 6, 1928. Appl. Dec. 6, 1923.

This invention relates to a continuous process for the refining of hydrocarbon oils such as petroleum products, tar oil, brown tar oil, and the like. It relates especially to a continuous catalytic process for the conversion of these hydrocarbon oils into lower boiling products, as in the production of gasoline from petroleum. Conversion of hydrocarbon oils into lower boiling products can be more effectively accomplished by the use of a catalyst, and at lower temperatures and pressures than by ordinary cracking processes. The catalyst found to be most satisfactory in this process is tin, or an alloy of tin containing a small percentage of antimony. The catalytic decomposition of the oil is most efficient when the oil is introduced into the body of molten catalyst in the form of a fog of oil suspended in a hydrogenous gas. By this means the oil is subjected to successive decomposition and hydrogenation in the presence of hydrogen, whereby saturated hydrocarbons of

lower boiling points are produced with a minimum loss due to the formation of coke and gas. The temperature of the alloy of tin and antimony is maintained between 350° and 450°C., while conversion of the oil to be treated take place in the liquid phase. H. E. T.

453. Refining of Petroleum (with hydrogen fluoride). F. Hofmann and W. Stegemann. E.P. 292,932, May 23, 1929.

Petroleum is refined by treatment with hydrogen fluoride, with or without the addition of metal halides, at ordinary or increased temperatures and/or pressures.

Thus a petroleum fraction, b.p. 150—220°, which tends to precipitate an excessive amount of asphalt, is treated for 2 hrs. at the ordinary temperature with 3 per cent. of hydrogen fluoride; the refined product remains as clear as water and has lost its unpleasant odour.

A crude petroleum fraction, b.p. 50—225° at 3 mm., having bromine number 2·2 is treated at room temperature with 4 per cent. of hydrogen fluoride. After 1, 2, 3 and 4 hrs. the bromide number drops to 0·68, 0·15, 0·09 and 0·06 respectively. The same fraction, after treatment for 4 hrs., with 1, 2 or 8 per cent. of hydrogen fluoride, has bromine numbers 0·56, 0·26 and 0·07 respectively. The same fraction, treated for 4 hrs. with 4 per cent. of hydrogen fluoride at 0°, 25° and 50° has bromine number 0·50, 0·09 and 0·08 respectively. The same fraction, treated for 4 hrs. at room temperature with 4 per cent. of hydrogen fluoride and 2 per cent. of boron fluoride, has bromine number 0·01. W. S. N.

454. Refining of Oils Derived from Coal (with Hydrogen Fluoride). F. Hofmann and W. Stegemann. E.P. 292,933, May 23, 1929.

Oils derived from coal are refined by treatment with hydrogen fluoride, with or without the addition of metal halides, at ordinary or increased temperatures and/or pressures.

Thus a crude benzene having a washing loss of 9 per cent. is treated for some time with 3 per cent. of hydrofluoric acid. The latter is then removed by careful heating and may be used for immediately treating a second batch of benzene. The refined benzene is distilled and then has a washing loss of 0.2 per cent. and scarcely shows any sulphuric acid reaction. W. S. N.

455. Refining Hydrocarbon Oils. H. K. Ihrig. U.S.P. 1,712,619. May 14, 1929. Appl. June 15, 1927.

The patent describes a method of treating normally liquid hydrocarbons in the gaseous phase by bringing the vapours into intimate contact with a properly heated mixture of hydrated lime and iron oxide in approximately the proportion of one to three. The process is stated to effect a substantial reduction of sulphur and nitrogen compounds present in the raw material. W. H. T.

456. Manufacture of Carburetting Liquids Similar to Petrol. E. A. Prudhomme, Assr. to Soc. Internationale Des Procedes Prudhomme (S.I.P.P.), Paris. U.S.P. 1,711,855, May 7, 1929. Appl. Jan. 20, 1925. Fr., May 18, 1924.

Hydrocarbon products are distilled in a current of hydrogen containing gas which is passed through the residuum of a previous distillation and then through the products in the course of distillation. The combined gases and distilled products are purified and then subjected to a stayed catalysis in a series of catalysers charged respectively with nickel at a temperature

of 180—200° C., then nickel and vanadium at 200—250° C., and finally nickel and cobalt at 180° C., in order to effect successively a general hydrogenation of the combined products, a splitting up of the hydrogenated products into unsaturated hydrocarbons with liberation of hydrogen and a saturation and polymerisation of the unsaturated products. The temperature throughout the apparatus is maintained sufficiently high to avoid condensation of the heavier fractions, and the product from the final catalyser is led to the usual type of condenser. H. S. G.

457. Manufacture of Natural Gasoline. H. E. Thompson, Assr. to Carbide & Carbon Chemicals Corporation. U.S.P. 1,695,162, Dec. 11, 1928. Appl. April 12, 1924.

The process provides for the manufacture of natural gasoline, by means of producing the gasoline under pressure and at a corresponding temperature, then cooling the gasoline below its normal boiling point by withdrawing vapours from a confined space containing the gasoline, compressing, cooling and expanding those vapours and returning the cold expanded material to the space containing the gasoline. S. W. C.

Kerosine.

458. Process for Decolorising Kerosene, Gasoline and Similar Petroleum Distillates. V. A. Kalichevsky. Assr. to Union Oil Co. U.S.P. 1,695,251. Dec. 11, 1928. Appl. Sept. 19, 1927.

The invention provides a process for the purification of petroleum distillates containing difficultly adsorbable colouring matters, comprising subjecting the stock to a caustic soda wash sufficient to convert colouring matters into adsorbable forms, then subjecting the distillate to the action of acid and clay to adsorb the converted constituents, and removing the clay from the distillate to yield a final product, these steps being carried on continuously. S. W. C.

459. Method of Refining Mineral Oils. A. Lachman. U.S.P. 1,712,960. May 14, 1929. Appl. May 18, 1926.

This invention relates to a method of refining mineral oils, kerosine or gasoline. The method consists in first treating the crude mineral oil, with sulphuric acid, at a temperature above but approaching atmospheric temperature. The oil is decanted from the acid sludge formed, and the remaining acid in the oil is neutralised with alkali. The oil is now heated to a higher temperature, and treated with phosphoric acid. Finally the acid sludge is removed by decantation and any remaining acid is neutralised with solid calcium hydrate, while suspended solids are removed by filtration. The following example illustrates the process. (a) One hundred parts by weight of crude lubricating oil were treated with four parts by weight of 66° sulphuric acid, the temperature being 45°C. After standing overnight the oil was decanted from the heavy acid sludge, and heated with three parts by weight of phosphoric acid of 85 per cent. strength. The oil was heated to 170°C. and the treatment continued for two hours. The mixture was mechanically stirred during this period and air was excluded. After cooling, the oil was decanted from the sludge and washed with dilute sodium hydroxide and finally with water. The oil was finally dehydrated by air blowing and filtered. This oil showed an increase in colour during the six hours of heating of 8 per cent., and has therefore a much higher stability than oils which have been clay treated. H. E. T.

460. Fractional Distillation. J. Primrose. U.S.P. 1,707,369. April 2, 1929. Appl. Jan. 30, 1924.

This invention consists in an improved method of distillation, especially adapted for use in the distillation of mineral oil. The general object is to provide a method of fractional distillation which can be carried out with a desirable heat economy, and which will yield clean cut fractions without any cracking taking place. The oil to be distilled is circulated through a closed system, comprising a storage tank, and a fire heated tubular oil heater. The velocity of flow through the oil heater, is controlled relative to the heat absorption capacity of the heater, so as to gradually raise the temperature of the system without heating the oil to cracking temperatures. The different vapours successively liberated are withdrawn at higher temperatures as the oil is progressively heated up.

H. E. T.

Lubricating Oil and Wax.

461. Process of Refining Used Oils. R. V. Aycock and W. D. Harris, Assrs. to Refinoil Manufacturing Corporation. U.S.P. 1,707,671. Apr. 2, 1929. Appl., July 27, 1928.

The process provides a method of refining used mineral lubricating oil containing water and hydrocarbon diluents which consists in removing free water from the oil, heating the oil to approximately 120° F., adding 2 per cent. by volume of 66° Bé. commercial sulphuric acid, agitating the mixture while maintaining it at said temperature, removing the oil from the resultant sludge, vaporising diluents from the oil by heating it to a temperature above 450° F. but below cracking temperature, adding to the oil while hot 15 to 18 per cent. by weight of fuller's earth, by causing the oil to pass upwardly through said fuller's earth, allowing the earth together with the products of reaction to settle into a bed, then drawing the oil downwardly through said bed, and finally drawing off the purified oil.

S. W. C.

462. Process of Imparting Fluorescence to Oil. J. C. Black, W. D. Rial and J. R. McConnell, Assrs. to Pan American Petroleum Co. U.S.P. 1,708,563. Apr. 9, 1929. Appl., Oct. 13, 1926.

The invention provides a process of imparting a desired green fluorescence to lubricating oil stock which does not contain the same, which comprises commingling the oil with an uncracked petroleum oil pitch made by a vacuum distillation of a petroleum oil which contains fluorescent bodies, at temperatures sufficient to extract from the petroleum oil pitch desired amounts of fluorescent bodies, and then separating the oil containing the desired fluorescent bodies from the petroleum oil pitch.

S. W. C.

463. Process for Making a Fluorescent Product. J. C. Black, W. D. Rial, and J. R. McConnell, Assrs. to Pan American Petroleum Co. U.S.P. 1,708,602. Apr. 9, 1929. Appl., May 6, 1927.

The invention provides a process for the manufacture of an extract of green fluorescent bodies, which comprises commingling a hydrocarbon oil with a comminuted coal tar pitch and a decolorising clay at temperatures substantially not less than 230° F., to extract from the coal tar pitch green fluorescent bodies, and then separating the hydrocarbon oil containing the green fluorescent bodies from the residual coal tar pitch and decolorising clay by filtration.

S. W. C.

464. Process and Apparatus for the Vacuum Distillation of Hydrocarbons. J. C. Black. U.S.P. 1,717,002. June 11, 1929. Appl., Feb. 2, 1924.

The invention provides a process for the production under reduced pressure of a reduced lubricating oil stock and the separation of an intermediate distillate and lower boiling hydrocarbons and water, from a mixture of the same, wherein the reducing and distilling operation is effected under a partial vacuum greater than 24 in. of mercury, which comprises circulating the mixture of hydrocarbons through heating tubes on to a pan in an evaporator, heating a portion of said hydrocarbons to a vaporising temperature and introducing steam therein, maintaining a reduced pressure on the evaporator sufficient to prevent decomposing or cracking of the hydrocarbons, vaporising the lower boiling hydrocarbons and passing the same together with the introduced steam into a fractionating tower, under reduced pressure, contacting said hydrocarbon vapours and steam in counter-current flow with a previously condensed portion of said intermediate distillate at a temperature sufficient to condense and extract from said mixture of hydrocarbon vapours and steam, substantially all the intermediate distillate free from water and substantially free from low boiling hydrocarbons, and separately condensing and collecting said lower boiling hydrocarbons and water. S. W. C.

465. Oil Reclaiming Process. C. Van Brunt. U.S.P. 1,713,200. May 14, 1929. Appl., April 1, 1925.

This application relates to the treatment of petroleum products containing suspended matter, with special reference to spent lubricating oil from automobile crank cases. To carry out the process, about 0.5 per cent. by weight of sludge, formed by the oxidation of mineral oils at high temperatures is first added to the oil to be clarified. Water-glass or a silicate of an alkali metal is now added in suitable amount, usually from 1 to 4 oz. to 1 gal. of oil. When thorough agitation of the oil with the water-glass has been effected, the mixture is brought into contact with a body of water, which is of sufficient bulk to take up the suspended matter. H. E. T.

466. Process for Making White Petrolatum. E. B. Cobb and A. Holmes. Assrs. to Standard Oil Development Co. U.S.P. 1,707,187. Mar. 26, 1929. Appl., Oct. 27, 1925.

The invention provides a process of preparing white petrolatum comprising dissolving crude petrolatum (10 parts by weight) in naphtha (90 parts by weight), adding fuming sulphuric acid to this solution, agitating, allowing to settle, withdrawing sludge formed by the acid reaction, repeating acid treatment several times, then extracting the petrolatum solution with a solvent (aqueous isopropyl alcohol) for sulphonic bodies, withdrawing the extracted solution, subjecting the extracted solution to a temperature sufficient to expel the solvent, and passing the petrolatum through a body of finely divided decolorising material. S. W. C.

467. Rectifying Lubricating Oil. M. Czarny and F. C. Frolander. U.S.P. 1,717,096. June 11, 1929. Appl., July 6, 1927.

This invention relates to oil rectifying systems which will maintain the quality of a lubricating oil constant during the operation of an engine, and especially an internal combustion engine. The apparatus, which is inserted in the oil circulation system, consists of a filter of a more or less conventional type and a rectifier which involves the novel principles claimed. The rectifier, for the removal of the liquid impurities, gasoline and water, consists of a

closed container for the oil. A tubular member carrying a wick is inserted through the wall of the container. The wick extracts liquid impurities from the oil by capillary action and carries them to an enlarged exposed portion lying outside the tube where they are evaporated. R. W. L. C.

468. Process of Refining Mineral Lubricating Oils. E. D. Gray, E. L. Scofield, and E. C. Defoe, Assrs. by Mesne Assignments to Standard Oil Co. U.S.P. 1,716,632. June 11, 1929. Appl., Nov. 9, 1925.

The invention provides a process of refining mineral lubricating oils which comprises treating the oils with sulphuric acid, settling and removing the settled sludge, then passing the oil without the addition of water thereto upwardly through a bed of cellulosic filtering material (*e.g.*, sawdust) substantially free from oil soluble resins (previously extracted), to remove the suspended sludge, and neutralising the filtered oil. S. W. C.

469. Method for the Treatment of Hydrocarbons, especially Oils by means of Corona Arc Discharges. W. Esmarch. U.S.P. 1,708,126. Apr. 9, 1929. Appl. Mar. 28, 1925.

An apparatus is described for treating hydrocarbons and especially oils by means of corona arc discharges. The apparatus comprises a source of high-frequency alternating current, with two electrodes connected to the source of current and an artificially-cooled jacket at one of the electrodes. A dielectric is arranged between the electrodes, in such a manner that it is separated from the cooled electrode by a narrow intermediate space, and from the other electrode by a wider intermediate space. H. E. T.

470. Lubricating Composition. G. W. Gray. U.S.P. 1,716,310. June 4, 1929. Appl. June 8, 1927.

This invention relates to lubricating compositions and more particularly to those for application to the rails of curved sections of railway tracks. For example, a lubricating composition consisting of a mixture of 60 per cent. of a cylinder stock residuum of approximately 200 secs. viscosity at 210° F. (Saybolt Universal) prepared from a paraffin or semi-paraffin base crude and 40 per cent. of a residual naphthene base oil of viscosity about 540 secs. at 122° F. (Saybolt furol) is very satisfactory at normal temperatures. The composition will have a viscosity of about 500–600 secs. at 122° F. (Saybolt furol) and a specific gravity of about 12–15° Baumé. R. W. L. C.

471. Apparatus for Refining Used Oil. F. O. Krieger. U.S.P. 1,707,673. Apr. 2, 1929. Appl., Oct. 24, 1927.

An apparatus is described for refining used lubricating oil, and it comprises a still, a receiver, a supply pipe entering the still, and a pipe joining the still to the receiver. A liquid actuated arrangement is provided for creating a vacuum in the still and receiver, and to draw used oil through the supply pipe. Diagrams of the apparatus are provided. H. E. T.

472. Process for Clarifying, Decolourising and Neutralising Oils. D. R. Merrill, Assr. to Union Oil Co. U.S.P. 1,695,198. Dec. 11, 1928. Appl., Feb. 1, 1926.

The invention provides a process of decolourising and neutralising oleaginous substances including petroleum lubricating oils, comprising commingling the oil with about 4 to 20 per cent. of raw clay together with water

in amount approximating half the weight of the clay and acid approximating ¼ per cent. of the substance treated, heating, separating the clay, agitating the oil with about 4 to 16 per cent. of a fresh amount of clay, adding a dilute alkali solution containing alkali less than ½ per cent. of the substance treated, heating and removing the clay. S. W. C.

473. Dewaxing Lubricants by Double Centrifuging. G. F. Olsen. U.S.P. 1,715,670. June 4, 1929. Appl., Oct. 15, 1927.

The invention relates to the removal of paraffin wax from lubricating oils which have been diluted with petroleum naphtha for the precipitation of the wax. The method consists in chilling and centrifuging the diluted oil in the following stages; chilling the solution at a rate not exceeding 4° F. temperature drop per hour to substantially 20° F.; centrifuging the chilled solution with a close adjustment of the machine for sharp separation of solution from wax. The oil-free wax is diverted from the system. The oil solution is further chilled to such a point as will bring the finished oil to the desired cold test. The second chilled solution is centrifuged with a slack adjustment of the machine to give a substantially wax-free oil which is diverted from the system and a slack wax containing some oil solution. The slack wax is added to a fresh batch of diluted oil which has not been chilled and the mixture is chilled and centrifuged under the conditions described above for the first stage of the process. R. W. L. C.

474. Wax-Bearing Vacuum Distillates. E. Petty. *Refiner*. 1929. 8 (6), 62-66.

The most recent method for the operation of pipe still and vacuum column equipment for the production of lubricant containing cuts consists in taking the wax distillate for cold filter pressing as an overhead cut which gives the highest degree of fractionation possible. The one side-drawn stream taken is prepared under proper vapour velocity conditions over from five to seven plates.

The heavy asphaltic liquid materials flow downwards from the vaporiser to the stripping section where entrained light vapours are recovered by a limited quantity of steam and carried into the upper portion of the column.

The side drawn stream receives the strippings by reflux from the overhead product so that its initial boiling point is closely regulated and there are enough plates in this service to pass all colouring matter of high boiling point into the base of the tower. This product has a maximum viscosity and the best possible colour with high flash point. The pipe still outlet temperature and the reduced pressure on the tower are so regulated that there will be no cracking of the colouring or pitch materials. This temperature varies from 715° to 790° F. depending on the characteristics of the crude.

A side drawn vacuum product taken under ideal temperatures and pressures from a properly designed tower will be of practically the same colour as a well-treated residue stock that has received treatment with 35–40 lb. of acid per barrel. The cold test will be high indicating that the wax is not dissolved or hidden in excessive colouring matter or solvent material.

The colouring matter that is present is of the same boiling range as the wax-lube material and in the proportions to produce amorphous instead of crystalline wax. When such a wax distillate is blended with 60 per cent. of naphtha the entire mixture is intersoluble at 100° F. On chilling the solution to 0° F. the wax or petrolatum phase contains only about 40 per cent. of naphtha so that more than 60 per cent. of naphtha will be found in the oil solution.

This change in distribution of naphtha does not occur when oil carrying crystalline wax is diluted with naphtha and chilled and the wax phase will then be found to contain nearly the full 60 per cent. of naphtha.

It is this change in solubility that accounts for the fact that amorphous wax can be centrifuged and crystalline waxes cannot. The rearrangement or redistribution of the naphtha sets up two components of different density, that is wax-naphtha component (60 per cent. wax, 40 per cent. naphtha) and an oil-naphtha component (40 per cent. oil, 60 per cent. naphtha). Naphtha is a solvent for waxes at ordinary temperatures and a precipitant at reduced temperatures. When wax-oil-naphtha solutions containing 60–70 per cent. naphtha are cooled there is a peak of crystallisation between 50° and 30° F. At about 30° F. the wax particles throw off naphtha. By continued chilling the first wax continues to give off naphtha and becomes more firm and at between minus 30 and minus 40° F. another saturation point is reached and the remainder of the wax will be precipitated.

With more selective precipitant-solvent materials the waxes can be precipitated at a much higher temperature and in a more definite form, but no solvent is as yet available which can compare with naphtha on a commercial and economic basis.

A vacuum-distilled stock of from 90–150 secs. viscosity has been produced from Mid-Continent black fuel bottoms, which can be dewaxed by centrifuging without further treatment after blending with 65–70 per cent. of naphtha and chilling to minus 40° F. After treatment and removal of naphtha a finished lubricating stock of low cold test and of cloud test below zero results. At the same time during the original distillation, a directly pressable wax distillate is obtained from the top of the tower and asphalt from the bottom stripping section.

Some preliminary work has shown that satisfactory dewaxing can be carried out by the filter aid method which possesses the advantage over centrifuging that less diluting material is required and equal dewaxing effects can be obtained at higher temperatures. R. W. L. C.

475. Process for Distilling Mineral Oil. A. E. Pew and H. Thomas. U.S.P. 1,714,811. May 28, 1929. Appl., May 29, 1926.

A process and apparatus are described for fractionally distilling mineral oil by heat exchange between mercury vapour and the oil to be distilled, the oil being partly vaporised and the mercury vapour being condensed and returned to the main body of liquid mercury which is continuously generating mercury vapour.

A stream of oil flows through a series of confined spaces, in the form of a rapidly flowing solid stream of uniform width and thickness. The mercury vapour generated in the mercury boiler is at such a pressure that it has a temperature above that of the hottest part of the stream of oil, this being the oil temperature in the last space of the series. The mercury vapour flows in chambers arranged in multiple with respect to each other, and in heat exchange relation with the respective confined spaces through which the oil flows. The amount of mercury vapour admitted to each chamber is sufficient to effect, by heat exchange and condensation, a partial vaporisation of the oil. It is desirable to maintain a minimum temperature difference between the oil and mercury vapour, in the several vaporising units.

H. E. T.

476. Dewaxing of Oil. E. B. Phillips and J. G. Stafford, Assrs. to Sinclair Refining Co. U.S.P. 1,714,133. May 21, 1929. Appl., Feb. 19, 1924.

The invention provides an apparatus for dewaxing oils by means of admixing a small amount of finely divided solid material with the oil to be dewaxed,

and simultaneously agitating and chilling the mixture and subsequently subjecting the chilled mixture to a filter pressing operation. The apparatus comprises an insulated tank having refrigerating coils and agitating means therein, means for supplying oil to be dewaxed and means for supplying a finely divided solid material to said tank, a filter press with a connection for charging the mixture from the tank to the filter press, said filter press being adapted for filtering wax and finely-divided solid material from the oil under pressure, a melting tank adapted to receive the wax and finely-divided solid material from the filter press and permit the separation of the wax from the finely-divided solid material, a dryer adapted to receive the finely-divided solid material separated from the wax in the melting tank and means for returning dried finely-divided solid material from said dryer to the insulated tank.

S. W. C.

477. Process of Purifying and Adding a Green Fluorescence to Lubricating Oil. W. D. Rial and W. R. Barratt, Assrs. to Pan American Petroleum Co. U.S.P. 1,708,592. Apr. 9, 1929. Appl., June 7, 1927.

The invention provides a process of purifying and improving the quality of lubricating oil containing acid constituents comprising commingling the lubricating oil with substantially dry calcium hydroxide and a coal tar pitch containing green fluorescent bodies at a temperature of substantially not less than 230° F. in quantities sufficient to neutralise and adsorb the acid constituents contained therein and add green fluorescent bodies to the lubricating oil and then separating the coal tar pitch residue and excess calcium hydroxide containing the adsorbed calcium salts from the lubricating oil and green fluorescent bodies and finally clarifying and decolorising the lubricating oil to the required degree with a clay.

S. W. C.

478. Process and Apparatus for Reclaiming Waste Oil. R. R. Rosenbaum. U.S.P. 1,708,488. Apr. 9, 1929. Appl., June 1, 1927.

The object of this invention is to produce a simple and efficient process for removing the impurities from used crank case oil or transformer oil. A further object of the invention is to recover the gasoline with which the used oil becomes diluted in a pure state.

The used oil is first subjected to treatment in the presence of heat and vacuum and under agitation, with an alkali and an oxygen supplying agent. The alkali used may be sodium silicate and the oxygen supplying substance could be a per-salt, such as per-carbonate, per-borate or per-sulphate. The mixture is heated to 200° F. when the alkali will saponify any animal, vegetable or fish oil, while the liberated silicate forms a gel which will absorb the soap and many other impurities from the crank case oil, such as carbon colouring matters and dust.

The oil remaining after the lower layer of sludge obtained in the above saponification process is drawn off, is treated with fuller's earth impregnated with sulphur dioxide. The oil is now heated up under vacuum to 400° F., when all the gasoline will be drawn off as a water-white liquid, which can be used for ignition purposes.

The oil now remaining is filtered off from the fuller's earth and is obtained in a new condition ready to be used again for lubricating purposes. A description of the apparatus used for carrying out this process is given together with diagrams. H. E. T.

Special Products.

479. Sulphonated Oxidation Products of Petroleum as Insecticide Activators. M. T. Inman. *Ind. Eng. Chem.* Vol. 21, **6**, 542-543.

The author has found that an increase in the efficiency of various insecticides is brought about by adding oxidation products of Pennsylvanian gas oil to the insecticide. A great number of samples have been made for field tests in this research. Emulsions of oxidation products of various boiling-point ranges and the addition of chemical groups have provided a great variety of results. The product formed by the following method was found to be the most satisfactory.

A straight run distillate of 38° Beaumé gravity from Pennsylvanian crude oil was oxidised by the Jawes catalytic vapour phase oxidation process. The condensate obtained consists of a mixture of alcohols, aldehydes, ketones, saturated and unsaturated acids, together with unchanged hydrocarbons. Sulphuric acid was now added slowly to the oxidised gas oil, the mixture being well cooled. The reaction mixture was then decomposed with water, which hydrolysed a large percentage of sulphonates and all sulphates, and then washed with sodium hydroxide. The final product contains about 40 per cent. of oxygen and 1·5 per cent. of sulphur. The activator itself is a clear maroon coloured liquid, easily miscible with water and possessing a high toxic value. H. E. T.

480. Making Asphaltic Products. L. Kirschbraun. U.S.P. 1,715,069. May 28, 1929. Appln., Jan. 27, 1923.

A process is described for making oxidised asphalt in which petroleum residue is passed together with air through an elongated conduit where the temperature of the mixture is gradually raised. The material is passed in the form of a spray from the conduit to the bulk supply tank, the contents of which are subjected to a sustained agitation. Liquid oxidised asphalt is continually withdrawn from the bulk supply. W. H. T.

481. Determining Value of Pipe Coatings. K. H. Logan. *Oil and Gas J.* 1929, **27** (50), 33, 87, 90, 94.

Three characteristics are necessary for a pipe coating—it must prevent corrosion, be practical as to application, and be sufficiently inexpensive.

Recent studies of corrosion causes tend to show that when a line passes through two different soils a galvanic cell is set up and corrosion occurs at the anode. To test pipe under field conditions requires considerable time and a more rapid method is required. Accelerated tests are of two classes —those in which severe conditions are imposed and those in which conditions are normal and failure detection more than usually sensitive. Since dry bitumens have a high specific resistance the change in resistance on exposure to moisture will indicate absorption of moisture and therefore deterioration of the coating. A series of tests were started by the Bureau of Standards. The central portion of the coated pipe was exposed to moist soil and the current produced by a dry cell measured by a micrometer. One pole of a dry cell is connected to a bare spot in the pipe and the other to a galvanometer or milliammeter. The other terminal of the galvanometer is brought into contact successively with columns of clay contained in a rubber pad strapped over the surface of the coating, the pad having ¼ in. holes 1 in. apart in which the clay is placed. Where the earth is in contact with a pinhole or crack in the coating the circuit is completed and the galvanometer deflected.

The determination of the resistance essential for a satisfactory coating requires further work and methods of determining the corrosiveness of soils are also being determined. G. S.

482. MANUFACTURE OF ASPHALT EMULSIONS. W. H. Schmitz. U.S.P. 284,330, April 23, 1929. Appl., Jan. 24, 1928.

The invention claims a method of producing bituminous emulsions consisting of mixing the bituminous substance with an alkaline slime. The bitumen is that obtained from waste liquors of lubricating oil refining, while the slime contains about 1 per cent. of bleaching earth and an alkalinity of about 0·3 per cent. The bitumen and slime are agitated vigorously in order to produce a suitable dispersion and a durable bituminous emulsion.
W. H. T.

483. THE CHARACTER, PROPERTIES, AND POSSIBLE USES OF BENTONITE. R. M. Woodman and E. McKensie Taylor. *J. Soc. Chem. Ind.* 1929, **48** (22), 121T.

Bentonite is an end product of the alteration of volcanic ash. The latter is supposed to have been porous and to have contained various gases. As a result of auto-metamorphism, the clay-like mineral composing Bentonite was formed. It appears to consist of two portions, firstly unaltered mineral felspars, biotite, etc., and secondly a clay portion containing 73-86 per cent. of pure Bentonite. It is colloidal in character and has been proved to consist mainly of sodium clay exhibiting the usual properties such as liberation of sodium hydroxide by hydrolysis in water, impermeability to water, etc. It has been proved a good emulsifier for certain oils, usually forming the oil-in-water type of emulsion suitable for spraying plants. In the case of cresylic acid, it has been shown to give the dual types of emulsion. In addition, Bentonite is to some extent a water softener acting by base exchange, this being useful in cases of permanent hardness. The fault of the material as a practicable emulsifier and water softener is its impermeability to water.
W. H. T.

Analysis and Testing.

484. REGENERATION OF CATALYSTS. H. S. Hirst, S. W. Rowell and Imperial Chemical Industries, Ltd. E.P. 309,888. Apr. 17, 1929.

In the manufacture of acetaldehyde from acetylene in the presence of mercury salts activated with, *e.g.*, salts of iron, sludge is deposited consisting principally of reduced mercury, but containing also, *e.g.*, iron. It is proposed to recover the mercury-iron catalyst from the sludge by dissolving the latter in strong nitric acid, if necessary with the aid of heat, and treating the solution so obtained with strong sulphuric acid to precipitate the whole of the mercury (and iron) (this occurs when the solution reaches a density not less than 1·45, the concentration of sulphuric acid being then about 55%), separating the precipitated sulphates and redissolving them in dilute sulphuric acid (15—20%). The filtrate from the sulphates, consisting of nitric and sulphuric acids, is recovered for further use by distillation. W. S. N.

485. CALCULATING THE SPECIFIC HEATS OF HYDROCARBON VAPOURS. W. K. Lewis and W. H. McAdams. *Chem. & Met. Eng.*, Vol. 36, 6, 336.

The following equation is given, as being the best available for calculating the specific heats of paraffin hydrocarbon vapours at low pressures:—

$MCv = 4{\cdot}4 + 4{\cdot}4n + (0{\cdot}012 + 0{\cdot}006n)t$ where t is degrees C.

$MCp = 4{\cdot}19 + 4{\cdot}29n + (0{\cdot}0067 + 0{\cdot}0033n)t$ where t is degrees F.

In these expressions n is the number of carbon atoms for molecule and MCp is the mola rheat capacity, expressed as B. Th. U. per lb. mol. per degree Fahrenheit.

The above equation is applicable to mixtures of paraffin hydrocarbons, providing the average value of n for the hydrocarbon mixture is employed, as may be determined by ordinary vapour density measurement. H. E. T.

486. Method for Determining Vapour Densities at Room Temperatures. E. F. Linhorst. *J. Amer. Chem. Soc.*, 1929, **51**, 1165-1167.

Two two-litre round-bottomed flasks, the upper one of which is inverted, are fitted with rubber stoppers and connected by an oil manometer, made of glass tubing 5 mm. inside diameter. The lower arm of the manometer tube is sealed and projects 4 ins. into the lower flask, a small hole being blown just below the stopper. The liquid whose vapour density or molecular weight is to be determined is sealed up in a Victor Meyer vapour density bulb, and hung in a loop formed by one end of a copper wire coil wrapped around the sealed end of the manometer tubing. Each flask is fitted with a stopcock connected to a mercury manometer and vacuum pump. The evacuating tube projects into the lower flask and is bent at right angles so that it will crush the bulb when turned from the outside.

About 0·1 gm. of liquid is employed, and the stopcocks are closed after evacuating the flasks to about 1 cm. of mercury pressure. The bulb is then crushed, and when evaporation is complete the increase in pressure is noted from the oil manometer. The temperature is read, and volume of lower flask must be determined. The gas equation $PV = WRT/M$ can now be solved for M. The volume of the lower flask is made more uniform for different determinations by grinding a shoulder on an over-size rubber stopper, whilst the error introduced by the working of the oil manometer may be neglected.

Liquids should have a vapour pressure of at least 4 cm. of mercury at the temperature of determination, otherwise a smaller sample must be taken. Good agreement has been obtained between this method and that of Victor Meyer. L. H. A.

487. The Determination of Unsaturated and Aromatic Hydrocarbons in Light Oils and Motor Spirits. A. B. Manning. *Jour. Chem. Soc.*, 1929, May, pp. 1014-1020.

This paper presents an account of a new method of determining the percentages of unsaturated and aromatic hydrocarbons in light oils and motor spirits. The method consists in passing a small quantity of the oil vaporised in a current of air, through a suitable reagent, the increase in weight of the latter being determined. A convenient piece of apparatus consists of a stoppered U-tube, with two potash bulbs in series. The oil is weighed into the U-tube and the bulbs are charged with the absorbing liquid. A gentle current of air free from carbon dioxide is then passed through the apparatus until all of the oil has been vaporised, and the absorption bulbs have attained a constant weight. Under the conditions employed, an average sample of light oil (0·20-0·50 grams) requires from two to eight hours for complete evaporation. To avoid difficulties due to non-volatile residues, the oil is redistilled before a determination is made.

I.—Determination of Aromatic Hydrocarbons in Mixtures containing no Unsaturated Hydrocarbons.

Preliminary experiments on mixtures of this type indicated that 98 per cent. or even 100 per cent. sulphuric acid failed to absorb benzene and toluene completely, the 98 per cent. acid taking up only about 70 per cent. of the toluene present. Complete absorption of the aromatic hydrocarbons occurred

when fuming sulphuric acid containing 5 per cent. of SO_3 was employed, but this reagent attacked the unsaturated hydrocarbons. Addition of 2-3 per cent. silver sulphate to 98 per cent. acid was found to greatly accelerate the sulphonation of benzene and its homologues, without producing any effect on the action of the acid on the saturated hydrocarbons. Under the experimental conditions employed there is always a slight absorption, varying from 0·10-1·0 per cent., of the saturated hydrocarbons, but the use of the second absorption bulb allows a correction to be made for this error. Thus in a synthetic mixture containing 16·3 per cent. of aromatic hydrocarbons, the absorptions in the two sets of bulbs were 17·15 and 0·80 per cent., giving a corrected figure of 16·35 per cent. for the aromatic hydrocarbons.

II.—Determination of both Unsaturated and Aromatic Hydrocarbons in Hydrocarbon Mixtures.

Sulphuric acid of 80 to 90 per cent. strength was found incapable of removing unsaturated hydrocarbons completely without attacking the aromatic hydrocarbons. The procedure finally adopted was to absorb the unsaturated and aromatic hydrocarbons together in a suitable nitrating mixture (10 per cent. concentrated nitric acid, or 16 per cent. of potassium nitrate in sulphuric acid). Absorption of unsaturated and aromatic hydrocarbons is complete, without any polymerisation occurring. To determine the aromatic hydrocarbons separately they are isolated in the form of their nitro derivatives. The weight of nitro derivatives obtained, multiplied by the experimental factor 0·452, gives the weight of aromatic hydrocarbons present in the oil under analysis. During the nitration the unsaturated hydrocarbons are oxidised to carbon dioxide, water and presumably acidic bodies which are soluble in water or aqueous sodium hydroxide. A more accurate value for the percentage of aromatic hydrocarbons is obtained by fractionating the oil through an efficient column, fractions being taken at 95°C. and 124°C. The aromatic hydrocarbons in the three fractions are then determined separately, appropriate conversion factors being employed. The nitro compounds may also be estimated volumetrically with titanous chloride. H. E. T.

488.—Determining Gasoline Content of Gas. M. Shepherd. *Oil and Gas J.* **28**, 6, (48, 170–178).

This article is a resumé of the detailed technique of the method of the Bureau of Standards Journal of Research for June, 1929, in which the accurate determination of gasoline in natural gas is described.

Natural gas is composed chiefly of saturated hydrocarbons of which proportion ordinarily making up the condensate called "natural gasoline" may be of the order of 1—2% of the total gas. The vapour pressure is a definite criterion. The "natural gasoline" content is accordingly defined as the hydrocarbon condensate derived from natural gas which is composed of the total percentages of pentane together with higher boiling hydrocarbons occurring as vapours in natural gas, plus sufficient percentage of normal butane to cause the total condensate to have a specified saturation pressure p at a specified temperature T. In essence, the method consists in specially separating natural gas by fractional distillation at low temperatures, the resultant fractions being then recombined in such a way that it is possible to determine the maximum amount of hydrocarbon condensate present in the original gas which has any given vapour pressure at any given temperature.

In the method p was determined over a relatively wide range keeping T constant (100°F), and also p determined for various values for T. The sample of natural gas was separated in a special form of apparatus by isothermal

fractional distillations into 3 factors (1) isobutane, propane and hydrocarbons of lower boiling points together with some N_2, O_2, CO_2 and rare gases, (2) normal butane and (3) pentanes and hydrocarbons of higher boiling points. Various curves are given showing the vapour pressure of mixtures of pentane and butane fractions. A comparison is made with the existing field and laboratory methods such as the compression cooling method, oil absorption method and the charcoal adsorption method. W. S. C.

489.—The Shorto Viscometer. *Journ. Scientific Instruments.* 1929, **6**, 199.

The instrument is introduced as an auxiliary to the Redwood Viscometer, and its results are capable of prompt conversion into Redwood degrees. The main features may be outlined as follows: Regulation of temperature. By a simple device, this is rendered automatic and the temperature will remain constant to within $\frac{1}{4}$°F. for an indefinite period.

Three sizes of jet. There are three oil cups and jets, one each for heavy, medium and light oils. Against each oil cup a factor is engraved and multiplication of the results by this factor expresses them in Redwood degrees. Facility for cleaning. The bottom of each cup (or rather tube) is composed of a tapered plug with a sharp convexity at both top and bottom. Through this convexity the jet is drilled vertically so that the orifice itself is placed well above the bottom of the tube. At the conclusion of each test it is only necessary to lift the plug by the rod attached, when it can be held up to the light and the jet inspected. The tube itself is left clear and without obstruction, and the whole operation of cleaning can be performed in less than a minute. The quantity of oil required for the test is only 35 ccs. W. H. T.

Refinery Plant.

490. Treating Hydrocarbon Oils. G. B. Bogart. U.S.P. 1,709,304. Apr. 16, 1929. Appl., Feb. 17, 1927.

A process is provided for carrying out a fractionating operation in such a way as to enable the production of volatile distillates such as gasoline, substantially free from undesirable higher boiling fractions. A rapid rate of distillation is made possible without affecting the quality of the distillate obtained, while the capacity of the fractionating equipment is increased in order to obtain increased yields of properly fractionated distillates. A further object of the process is to control the cooling water to the tower in such a way that the amount of cooling may be controlled without affecting the volume of liquid supplied. Furthermore, a portion of the condensed vapours is returned to the fractionating column in order to assist in obtaining the correct reflux ratio, and to produce close fractionation of the vapours. H. E. T.

491. Distilling Liquid Mixtures. D. G. Brandt, Assr. to Heat Treating Co., N.Y. U.S.P. 1,713,254, 1,713,255 and 1,713,256. May 14, 1929. Appl., Mar. 24, 1920. Divided Apr. 8, 1921 and Sept. 24, 1926.

Crude petroleum to be treated is supplied by means of a pump and inlet pipe to the bottom of a still, and passes in a continuous stream through a series of combined heat recovery and fractionating chambers in a counter-current direction to, and in a heat interchanging relation with hot vapours and liquids produced in a later step of the process, and which are distributed throughout the oil in each chamber by means of inverted perforated trays so arranged that the oil flows through serrations on the lower edge of the tray

while the vapours and gases pass upwardly through perforations in the horizontal surface of the tray. The residual oil is finally heated to a still higher temperature, and an additional quantity of oil is vaporised in a boiler or still directly fire-heated. During the final heating and vaporisation of the oil, gas and vapours are bubbled through the heated oil to promote uniform heating, and the additional vapours produced are absorbed by the gas and carried out of the boiler. From the boiler the gas and vapours pass through a series of condensers placed in the fractionating chambers and are cooled to progressively lower temperatures by the inflowing stream of crude petroleum. A portion of the vapours is condensed in each condensing coil, the higher boiling fractions being condensed in the chambers receiving vapours in nearest communication with the boiler and progressively lighter fractions being condensed in each successive condenser. In passing successively through the condensers the gas is cooled by the crude petroleum and is deprived of substantially all of the petroleum vapours, and, after being still further cooled to remove the last traces of condensible vapours, is returned to the first fractionating chamber and mixed with fresh inflowing crude petroleum to be treated. To recover the heat remaining in the unvaporised residues remaining in the boiler and in the hot condensates formed in the condensers, the hot boiler residue passes from the boiler directly through the fractionating chambers parallel to the condensing vapours and the condensate from each condenser is also passed through succeeding cooler chambers before leaving the apparatus.

H. S. G.

492. Art of Distilling Oils. E. M. Clark and F. A. Howard, Assrs. to Standard Oil Development Co. U.S.P. 1,710,404. Apr. 23, 1929. Appl., Dec. 24, 1920.

The invention provides a process of distillation which consists in subjecting a plurality of bodies of distillate oil of about 32° B. at 4 to 6 atm. pressure to a temperature suitable for the pyrogenetic distillation of gasoline therefrom, passing the vapours thereform through bodies of oil, thereby heating the latter and condensing part of the vapours, flowing streams of like oil through coils in the last mentioned bodies of oil at about 650° F., collecting the oil from said streams and utilising it as charging stock, passing the vapours from the last mentioned bodies of oil through additional bodies of oil, thereby heating the latter and condensing a further portion of the vapours, flowing oil of lower specific gravity through coils in the last mentioned bodies of oil and controlling said streams to maintain said bodies of oil at about 590° F. and feeding the heated oil from said streams into the distilling system during distillation.

S. W. C.

493. Converting Hydrocarbon Compounds. M. C. Colony, Assr. to Petroleum Conversion Corp., N.Y. U.S.P. 1,715,341. May 28, 1929. Appl., Jan. 3, 1927.

Oil is superheated without substantial cracking and the vapours passed to a non-externally heated reaction chamber containing a mass of inert refractory heat-absorbing material, and in which cracking is brought about by contact with a highly heated mixture of externally generated hydrocarbon gases and steam. A uniform temperature above the conversion point is maintained by the regulated admission of the relatively cold gas. The product from the chamber is led to fractionating and condensing arrangements of the usual character.

H. S. G.

494. Treatment of Mineral Oils. W. F. Downs and M. G. Downs. U.S.P. 1,716,372. June 11, 1929. Appl., Nov. 24, 1922.

A mixture of mineral oil and aluminium chloride is heated to a temperature sufficient to cause rapid evolution of vapours in a still provided with a lifting mechanism in the form of a screw propeller closely adjacent to the bottom for causing an upward circulation of solid matter, and a separate beater having blades, which have the effect of breaking up the masses resulting from the accumulation of tarry matter on the aluminium chloride particles. Both propeller and beater are driven by suitable external gearing. The resulting vapours are led to a receiving chamber above a filter bed composed of one or more layers of an inert material, *e.g.*, fuller's earth. Below the filter bed is another chamber where the liquid collects. The conduit leading from the still to the filter is provided with suitable heating means to maintain it above the vaporising point of aluminium chloride whilst the filter bed itself is cooled by water circulating in a surrounding jacket. If only decolorising and detarring is required without cracking the same apparatus may be used except that the reaction may be carried out in the cold, no vaporisation of oil and aluminium chloride taking place. H. S. G.

495. Apparatus for Treating Emulsified Oils. G. Egloff and H. E. Benner, Assrs. to Universal Oil Products Co. U.S.P. 1,694,643. Dec. 11, 1928. Appl., Aug. 21, 1920.

The process provides an apparatus for treating emulsified oils consisting of a shell still adapted to receive a bulk supply of oil having a vapour space therein above the oil level in said still, an elongated burner extending lengthwise of said still on each side thereof for producing heating gases discharging against the vapour space of the still to heat the surface of the oil therein to the maximum temperature, a mechanical agitator immersed in the oil in the still for agitating said oil during distillation, a condenser and receiver and means for conveying the vapours from said still to said condenser and the distillate from the condenser to said receiver. S. W. C.

496. Refining of Light Oil with Silica Gel at Rochester, N.Y. R. E. Fulreader. *Ind. Eng. Chem.*, 1929, **21**, 691-693.

Prior to 1927 the Rochester Gas and Electric Corporation used sulphuric acid in refining their light oil by-product (containing benzol, toluol, etc.) from the manufacture of coal gas and water gas. A final distillation was given prior to blending with ordinary motor spirit. The yield by this method was low, as valuable unsaturated compounds were removed together with the undesirable gum-forming constituents, and to overcome this the silica gel process was installed.

The plant was originally designed for continuously refining 4,000 gal. daily of 220–280° C. end point light oil (85 per cent. distilling at 180° C.). The oil to gel ratio was 25 to 1, but this has been increased to 200 to 1, and the oil throughout doubled.

The light oil is first intimately mixed with not more than 0·05 lb. conc. sulphuric acid (66° Bé.) per gal. of oil, in a Venturi mixer. The acid is necessary to remove moisture. The oil then passes to settling tanks from which it is drawn by the circulating pump. Sufficient pressure (100–175 lb./sq. in.) is maintained in the system to keep the oil in the liquid phase at the temperatures required. The oil then passes through a heat exchanger to a preheater where it is raised to 230° F., after which it passes upward through the percolator. This is conical in shape, has a central stirrer, and holds a 300 lb. charge of silica gel. A steam jacket maintains the temperature at 270° F. The percolator is of such capacity that the oil is in contact with the gel for

1 hr., being stirred constantly, at the end of which time the oil, now freed from gum-forming constituents, has reached the top of the percolator and is discharged through screened outlet pipes to the heat exchanger and cooler. The oil is then bubbled through caustic soda solution to remove all traces of acid, and given a final batch distillation to produce a motor benzol of 160° C. end point.

Silica gel, made by drying and crushing the jelly-like mass obtained by treating sodium silicate with sulphuric acid, owes its activity to its infinitesimal capillaries. The quality used in refining motor benzol is such that 80 per cent. passes a 250-mesh screen. A screw conveyer measures and feeds gel from a storage hopper into an agitator, where sufficient recovered oil is added to produce a mixture which will readily flow to the percolator. Here the 300-lb. charge can be used until the oil leaving the gel falls to a predetermined standard as to colour and gum content after distillation. When this point has been reached the preheated crude light oil is directed to a second percolator which has been charged with fresh gel.

The spent gel from the first percolator is then drawn off into the decanting hopper, fitted with outlet pipes at various levels. Clear oil is drawn off to the recovered oil tank, after which the gel is heated by the steam jacket, and open steam is blown through the mass to remove the greater part of the oil. The steam is condensed and the oil added to the recovered oil tank. The partially dried gel is then discharged by means of a bucket elevator into a hopper, from which it is fed with superheated steam to the primary activator. Here it is heated indirectly, by combustion gases from a gas-fired furnace, to 450° F., and the last traces of oil are distilled off, the distillate being added to the recovered oil. The gel is then taken to the secondary activator where it is heated indirectly to 550–600° F., the carbon, etc., being burnt off in a stream of air. Leaving the secondary activator the gel is raised by air draught to a cyclone box above the storage hopper, ready to repeat the cycle.

The plant has never shown an oil loss in excess of 3 per cent. The finished product has a colour of 19 to 25 Saybolt, a gum content (by copper dish) of 6 to 20 mgms., and is stable for a considerable period. Sulphur content is within specification limits, and the benzol is non-corrosive. The Rochester plant is run intermittently, which causes the cost of treating to be slightly higher than was the case with the old sulphuric acid process. This is more than offset, however, by the 15 per cent. increase in yield of motor benzol from the silica gel process. In future installations costs will be further reduced by means of a central activating plant run by the Silica Gel Corporation.

L. H. A.

497. Process for Distilling Oil. A. E. Harnsberger. E.P. 310,393. Appl. date, Jan. 23, 1928. Accepted April 23, 1929.

This invention provides a method of vacuum distillation of mineral oils based on the flow of oil through a heated pipe still, characterised by the fact that the pipe still forms a distillation zone having increasing cross sectional area or vapour space from the oil inlet end to the outlet end, where it is connected with a vaporiser or fractionating tower operated at low pressures.

These conditions are achieved by (1) constructing the pipe still with pipe of constant cross sectional area and providing a single pipe in the inlet unit of the still and a correspondingly increasing number of similar pipes arranged in multiple or parallel order towards the outlet of the still; or (2) constructing the still with pipe of constantly increasing cross section of gradually tapered form, or abruptly stepping up the diameter by use of ordinary pipe of different sizes. The increase in cross sectional area permits such reduction

in back pressure due to pipe friction of the oil and vapours as to allow the vacuum carried at the outlet end to penetrate back substantially to the inlet end. The length of the still and the velocity of the oil and vapours are so regulated that the oil and vapours are exposed to heat in the still for a period not in excess of ten seconds.

A complete vacuum-distilling plant for mineral oils embodying a pipe still of the above design, together with the necessary fractionating towers and auxiliary equipment, is described. R. W. L. C.

498. Prevention of Substantial Corrosion in Hydrocarbon-Oil-Treating Apparatus. J. C. Morrell and H. P. Beuner. U.S.P. 1,715,095. May 28, 1929. Appl., Sept. 3, 1925.

This invention has for its purpose the prevention of corrosion in vapour lines and condenser tubes of apparatus for treating hydrocarbon oils. A solution of ammonia in water is injected into the apparatus at a point where condensation of acidic water occurs. The ammonia solution not only neutralises the hydrochloric acid in the water but dilutes it owing to the excess of water.

As an example of carrying out the process the distillation of Smackover crude oil may be cited. This crude contains 2 per cent. of salt water, the salt being composed of magnesium and calcium chlorides which form hydrochloric acid on heating. By analysis it was estimated that 0·2 per cent. to 1 per cent. solution of ammonia would completely neutralise the hydrochloric acid formed during distillation. Fifty gallons per hour of this solution reduced the corrosion by neutralisation and dilution so that it was possible to operate the distillation unit over long periods without difficulty. H. E. T.

499. Refining Hydrocarbon Oil. E. H. Payne, Assr. to Standard Oil Co. of Indiana. U.S.P. 1,716,974. June 11, 1929. Appl., June 17, 1925.

A closed vertical filtering chamber has a constricted lower portion carrying a screen to support a body of filtering material, *e.g.*, fuller's earth. The filter is suitably mounted on a setting in which is provided a gas or oil burner playing on the lower portion of the filter slightly above the screen. In the lower portion of the filter is provided a plurality of perforated coils by which air or other oxidising gas may be supplied within the filter. Further closed coils extend throughout the mass of filtering material in the cylinder. In operation, oil is allowed to percolate through the body of the filtering material until the desired refining is no longer effected. The flow is then stopped and a suitable washing fluid forced into the filter. When the washing operation has been completed, the naphtha is removed by steaming and heat is then applied to the bottom of the constricted portion by means of the burner. Air is supplied through the perforated coils and ignition of the organic matter in the chamber takes place, the zone of combustion gradually travelling upwards through the filter controlled by the supply of air. Excessive heating is avoided by passing air or other cooling medium through the internal closed tubes. After combustion has ceased, the mass of filtering material is allowed to cool to any desired temperature and the filtering operation recommenced. H. S. G.

500. Distillation of Oils. A. G. Peterkin, W. F. Stroud, and R. B. Chillas, Assrs. to Atlantic Refining Co. U.S.P. 1,709,874. Apr. 23, 1929. Appl., June 4, 1925.

The invention provides a process of distilling petroleum or petroleum products without substantial cracking, which comprises rapidly heating

the oil to a temperature in excess of 750° F. without substantial cracking by passing it at high velocity through a zone in which heat is transferred thereto, maintaining the vapour in contact with the liquid oil while in the heating zone, immediately thereafter separating the vapour from the unvaporised oil, cooling the unvaporised oil to prevent cracking by bringing it into contact with steam in a zone substantially devoid of the separated vapour, immediately fractionating the vapour by passing it counter-current to a stream of reflux liquid oil and drawing off a portion of said reflux oil from contact with the vapour at an intermediate point in its course of travel.

It is claimed that by this means an asphalt bottom having a carbon residue less than 2·5 per cent. of the crude oil by Bureau of Mines test can be obtained.

S. W. C.

501. Process for Fractionally Distilling Hydrocarbons. R. T. Pollock, Assr. to Universal Oil Products Co. U.S.P. 1,710,068. Apr. 23, 1929. Appl., Apr. 11, 1921.

The invention provides a continuous process for treating hydrocarbon oil, comprising passing the oil in a stream of restricted cross-sectional area through a coil in a heating zone wherein it is highly heated, transferring the highly-heated oil from said stream into a reacting zone wherein conversion occurs, passing the evolved vapours to a dephlegmator, condensing and collecting the dephlegmated vapours, maintaining a substantial superatmospheric pressure on the oil undergoing conversion and the vapours in said dephlegmator, redistilling pressure distillate under a reduced pressure by the utilisation of the heat given up by the vapours passing through the dephlegmator and from products of combustion from the cracking still in a zone surrounding the dephlegmator, and returning reflux condensate from the dephlegmator together with raw oil and unvaporised pressure distillate to the inlet side of said coil to be passed there through for retreatment.

S. W. C.

502. Apparatus for Treating Oils. G. W. Wallace. U.S.P. 1,714,198. May 21, 1929. Appl., Oct. 24, 1924.

The apparatus consists of a horizontal or vertical closed reaction chamber, lined with firebrick and surrounded by insulating material which completely fills a steel shell. The chamber is partially filled with pieces of carbon or refractory material supported on a grate above an inclined plane which directs volatile products condensed in the chamber to travel to the outlet pipe. A surface combustion burner projects into the free space at the top of the chamber through an opening filled with refractory material. Concentric with the burner is a valve-controlled air inlet and atomising or injecting device situated so that the oil to be treated is preheated whilst passing through the inlet pipe. Means are provided for the introduction of hydrogenating vapours or gases and for carrying out the reaction under increased or diminished pressure.

H. S. G.

503. Pressure Distillation of Heavy Hydrocarbon Oils. R. E. Wilson, Assr. to Standard Oil Co. of Indiana. U.S.P. 1,712,187. May 7, 1929. Appl., June 29, 1925.

A drum still, mounted in a furnace setting, is connected by two large pipes, depending vertically, respectively, to upper and lower tube headers between which are provided inclined tubes also situated in the furnace setting. The still and associated tubes are filled with raw oil, preferably a distilled oil, which is brought to distillation temperature under a pressure

4–6 ats. Conversion to lighter products takes place and increase in asphaltene content of the unvaporised liquid takes place at a progressively increasing rate. After distillation has begun additional raw oil is fed into the system and oil is withdrawn from the still through a discharge line and valve so controlled with reference to the distillation system that the oil withdrawn has an asphaltene content not exceeding 0·4 per cent. and preferably in the neighbourhood of 0·1 per cent. The withdrawn oil is forced by a surge pump into and through a pipe still in which its temperature is increased by 100—150° F. under a pressure of 200—600 lb. sq. in., and at a rate sufficiently rapid to prevent carbon deposition from taking place and an additional conversion to lighter products not exceeding 6—8 per cent. of the oil passed through takes place. From the pipe still the oil passes into insulated enlarged drums, where it is retained long enough for an additional 25–40 per cent. conversion to take place. Unconverted oil is withdrawn through a discharge line and vapours pass into a reflux tower, the reflux formed there being discharged through a pump line into the line entering the pipe still. H. S. G.

Engineering.

504. Improvements in Temperature Indicating Devices. A. C. Spark Plug Co. E.P. 293,320. 1928.

A temperature indicating device consisting of a flexible member filled with a suitable mixture such as alcohol and carbon tetrachloride is fixed at one end to the cap of the radiator of an internal combustion engine. The free end of the member is coupled by means of suitable gearing to an indicator situated above the cap and also to a valve which is arranged to open or to close the water outlet branch from the engine as the temperature rises or falls. R. S.

505. Device for Cooling Packing. O. Beheimer, Assr. to Texas Oil Co. U.S.P. 1,716,162. June, 1929.

Shafts and piston rods used for stirring or agitating substances under high pressures and temperatures are packed to prevent leakage by means of a gland situated at the end of a tube remote from the portion of the apparatus at high temperature. The bore of the tube is small compared with the main bore and is of sufficient length to allow of substantial cooling of the gases before the gland is reached. The same device is applied to keep cool electrical insulators used for protecting conductors which are led into systems at high pressure and temperature. R. S.

506. Economic Fuel-Volatility and Engine Acceleration. Donald B. Brooks. *S.A.E. Journal*, June 24, 1929, p. 609.

This paper embodies an extension of the work described at the 1928 semi-annual meeting of the S.A.E. The tests then described have been co-ordinated with further work covering manifold temperatures from −10 to 100° C., and all results show a consistent variation in the acceleration obtained with the special test fuels as the manifold temperature is varied. The engine used is a six-cylinder overhead valve design with a carburetter arranged without accelerating well. The criterion for carburetter adjustment is the production of an effective air-fuel ratio sufficient to give acceleration with only occasional missing, and this results in the use of a mixture about 5 per cent. stronger than that required to ensure eventual acceleration. At low manifold temperatures the accelerations obtained are

in the reverse order of the 50 per cent. A.S.T.M. volatilities. Inspection of the distillation curves shows that the early fractions are not related to the 50 per cent. fractions as regards temperature of distillation. With increased manifold temperature the accelerations are in the direct order of the 50 per cent. point volatility and this order is maintained throughout, although the performance of all fuels gradually approaches that of aviation gasoline. Preheating the inlet air causes a still closer approach to aviation gasoline.

It is concluded that the effect of the 50 per cent. point on either quantity of fuel required or on acceleration is negligible except under summer operating conditions, and under these conditions, unless special devices are provided, the fuel required decreases, or the acceleration for a given supply increases, as the 50 per cent. point falls. It is probable that an increase in consumption would follow the raising of the 50 per cent. A.S.T.M. point.

R. S.

507. Volatility in Motor Fuel Performance. G. G. Brown. *Oil and Gas J.*, 1929, **27**, 42, 107.

Effective volatility is defined as the quotient obtained by dividing the weight of fuel in the mixture actually delivered to the cylinders of an engine by the weight supplied by the carburetter. These weights are only equal when the fuel is completely vaporised in the carburetter or lower part of the manifold. In practice sudden changes in pressure caused by throttle movements, and changes in velocity due to alterations of speed, cause variations between the weight supplied and the weight actually burnt. The apparatus used to determine the effective volatility consists of a standard overhead valve six-cylinder engine coupled to a dynamometer with an inertia disc arranged to reproduce the inertia effects of a car. The dynamometer is loaded to represent the rolling and wind resistance effects.

Commercial carburetters are unsuitable for the tests since they give mixture ratios at the choke tube which vary with the speed, and with the size of the accelerating well or pump; the fuel is therefore supplied from a burette fitted with a plunger which displaces fuel in proportion to the air supply, the plunger being operated from a gasometer through which the air required for the test is drawn. Change of speed during acceleration is determined from photographs of the pointer of an indicating tachometer taken at the rate of eight per second. Each test is started and stopped by a series of electric relays arranged so that contact is made at a definite point in the cycle, followed by starting an automatic stop watch, closing the revolution indicator circuit, opening the throttle, and starting the camera. The test is stopped electrically and all instruments thrown out of action. The engine is cooled by means of a closed system with a temperature control, and the manifold jacket can be heated or cooled to any desired degree. Arrangements are made so that the engine can be run on a standard carburetter or switched over to the air measuring gasometer and fuel feed burette as necessary.

The engine is first run for about two hours making accelerations on the standard cycle with carburetter feed and until check readings are obtained for about 18 minutes. The gasometer and displacement plunger are then coupled in and a few further tests are made, followed by a complete series with the camera. Each test with the displacement plunger is followed by one with the carburetter to enable any change of engine condition to be noticed by the observers.

Five fuels are reported on. These have A.S.T.M. distillations varying from 40° to 140° C. for 20 per cent. and from 90° to 200° C. for 80 per cent. The performance in each case is compared over a range of manifold wall

temperatures and air-fuel ratios with that of a fuel which is completely vaporised under the conditions of test. Curves from the richer mixtures can be duplicated almost exactly, but with weaker mixtures there may be no acceleration. Except with very weak mixtures the effective volatility of each fuel usually varies with the manifold temperature. Where the A.S.T.M. distillations show a differential between two fuels which changes sign during the distillation range the same effect may be noticed in the acceleration curves at low and high temperatures.

Under the conditions of test natural gasoline is 100 per cent. effectively volatile in blends with motor fuels. The difference between perfect and partial volatility is shown by the time lag in the acceleration curves at the lower end. The lag may vary from 0 up to 5 or 6 seconds, after which the slope of the acceleration line becomes almost constant except in cases of over-rich mixtures.

It is not possible at present to correlate completely the effective volatility of a fuel with the A.S.T.M. distillation characteristics, but it is estimated that about 25 per cent. to 30 per cent. of the fuel is carried into the cylinders in the form of mist, and that about 60 per cent. must be burnt for good acceleration. The effective volatility therefore depends on the A.S.T.M. distillation curve at 30 per cent. to 35 per cent. less than the 60 per cent. point. Owing to the use of accelerating wells and pumps the mixture supplied to a typical engine is stronger than the average during the early period of acceleration, and, in addition, most drivers demand instantaneous response to throttle opening. The net effect of the practical conditions is that the 30 per cent. point on the A.S.T.M. distillation curve is probably the most important point indicating engine performance during warming up in cold weather. If a strangler is used the 10 per cent. to 20 per cent. range is more important. When the engine is thoroughly warmed up the 60 per cent. to 70 per cent. range is probably the most important part.

The broad conclusions drawn are that the part of the A.S.T.M. distillation up to 40 per cent. to 50 per cent. is most significant in indicating the effective partial volatility of a fuel necessary for the easy starting and rapid warming up particularly desirable in cold weather, and that the 50 per cent. to 90 per cent. range indicates the effective volatility of the fuel under steady driving conditions after the engine has become thoroughly warmed up.

R. S.

508. Marine Oil Engines. Diesel Engine Users' Association. Sterry B. Freeman. Oct. 12, 1928.

The gross tonnage of oil-engined ships increased from one-quarter to three-quarters of a million tons between 1914 and 1919, and reached nearly five and a-half million tons by 1928.

In addition there are 280 vessels on order with an aggregate tonnage of over one and three-quarter millions.

The various types of engine in use and the different methods of drive are still under trial as regards relative merits, and it is not yet possible to standardise, as has been the case with steam machinery.

There has recently been a demand for higher powers which has been met in some cases by increasing the mean indicated pressure (made possible by improved design), and in others by increase of piston speed. The latter does not necessarily mean greater wear of liners, since this depends mainly on the R.P.M. and takes place at the top of the stroke when the piston is moving at the lowest speed. Power increase has also been made available, by driving compressors and scavenge pumps from auxiliaries, by developing the double-acting engine and by supercharging. Tabulated results from an

engine with the Buchi supercharging system show a possible increase of power of almost 100 per cent. Airless injection has also been further developed and waste heat utilisation is making progress.

The advance as regards material has been marked. The tensile strength, and elastic limit, of the steels in use have been increased, and special heat-resisting steels are to be had for exhaust valves and seats. Details such as telescopic water cooling pipes have received special attention, and may be of special bronze, cupro nickel or chromium coated brass.

It has been proved that cast iron in its pearlitic form is the only type suitable for resisting prolonged high temperatures, its strength being 50 per cent. greater than ordinary cast irons hitherto used. The extensive use of this material has involved the reorganisation of foundries on a more scientific basis.

The liners of the "Medon" (740 mm. bore) originally cast from "special" cast iron varied in rate of wear. One of the poorer ones had worn 0·166 in. at the top in five years, while one of the better ones only wore 0·102in. Piston slap developed 10 months after these figures were reported.

The liner wear on Doxford opposed piston two-stroke engine liners is quoted as 0·105 in. after three years' continual service.

The wear of liners varies down the stroke as the gas pressure in four-stroke engines, but it is more uniform to the top of the exhaust ports in two-stroke engines, and below the ports is small and of the same order as the four-stroke. Details of wear readings for several cases are tabulated.

Ring wear is greatest on the top three rings, and the up and down clearance of the first ring in an engine after two years' running was found to be 51-thousandths of an inch. The grooves wear almost twice as fast at the outer edge as at the inner. Proper fitting of new rings involves machining the grooves in the piston as well.

An alteration of rules for crankshaft sizes is in question. The wear down of main bearings has to be watched very carefully, owing to the nature of the stresses set up by small deflections. The usual clearance allowed in the main bearings is about 1/1000 in. per 2 in. of diameter. This is the minimum, but if it increases much, difficulty will probably be experienced in maintaining the requisite lubricating oil pressure.

Ease of overhauling depends to some extent on the weight of moving parts, and in this respect double-acting engines are about 50 per cent. heavier than single-acting of the same dimensions, although the number of cylinders is less for the same power.

The joints of piston cooling pipes require special care to avoid leakage of water if the pistons are water cooled. With oil cooling the risk of trouble is less.

It is necessary to keep the temperature of salt water cooling for pistons below 130° F. and to circulate at a high speed. With oil the circulation must be maintained after the engines have stopped to prevent carbonisation of the piston crowns.

The maximum probable difference in fuel oil consumption between a good and a bad engine is about 0·06 lb. per S.H.P. Hr., the range being about 0·38 to 0·44 lb. per S.H.P. Hr. Assuming a fuel bought at £3 per ton on a round voyage, the cost of this difference would amount to about £385 per 1000 S.H.P. per annum of 200 sailing days.

A table of lubricating oil consumptions shows variations from 4·42 to 10·88 gallons per day per 1000 S.H.P. including electric generators and auxiliaries. The high cost of lubricating oil may offset the low cost of fuel in certain cases.

Total fuel and lubricating oil costs per annum for four-stroke single-acting, two-stroke single-acting, four-stroke double-acting, two-stroke double-acting and the Scott-Still engine vary between £2958·6 and £3297·8.

Out of eleven ships under observation with four-stroke single-acting engines dating to five years old, and having a total of 148 cylinders, one cylinder head has been removed, one liner, two compressor H.P. pistons and no other parts except piston rings, springs, etc.

It may be taken as established that, unless unforeseen breakdowns and consequent renewals are experienced, the running costs of oil engines are not greater than those of steam engines. The oil engine is still expensive in first cost in comparison with the steam engine and requires careful handling, but the economy of space and weight, especially bunker weight, indicates that coalowners will have to do all they can if they are to keep up with the progress of the oil engine. R. S.

Anti-Detonation.

509. A Study of Auto-Ignition Temperatures. Henry James Masson and William F. Hamilton. *Ind. Eng. Chem.*, June 21, 1929. Pp. 544–549.

The authors refer to the work of Constam and Schläpfer, and Moore attempting to correlate auto-ignition temperatures with the suitability of fuels for given engines, especially those of the Diesel type. It is pointed out that Moore's results obtained from the "ignition meter" need equating to engine conditions by means of a constant determined for each case. With regard to knocking tendency of gasolines, Brown's equation is quoted :—

$$\frac{\text{Rate of pressure rise}}{\text{Absolute auto-ignition temperature}} = \frac{1}{\text{H.U.C.R.} \times \text{constant}}$$

Experimental curves are given to show the effect on the auto-ignition temperature of benzene due to blending with various percentages of carbon tetrachloride and diethyl ether. Carbon tetrachloride is not ignitible at 1000° C. nor will it ignite with 5 per cent. benzene, but the ignition temperature drops to about 585° C. with 10 per cent. benzene. This value is below that for benzene alone. Increasing concentration of benzene above 10 per cent. slowly raises the auto-ignition temperature.

It is pointed out that the use of carbon tetrachloride in circumstances which lead to concentrations of the order of 90 per cent. or less may give mixtures *more* inflammable than the other component of the blend. In the case of diethyl ether the minimum A.I.T. is about 480° C. at a concentration of 15 per cent. of diethyl ether in 85 per cent. benzene.

Other substances may have different characteristics, thus a second type of curve shows both a maximum and a minimum temperature for binary blends higher and lower respectively than the A.I.T.'s of the components. Ethyl Alcohol and either acetic acid or p-xylene illustrate this type. The curve for blends of n-heptane and iso-octane is regular and falls progressively from the A.I.T. of the iso-octane to that of the n-heptane, there is, however, no simple relationship between the tendency to knock in an engine and the A.I.T. of the fuel. Such figures as are available suggest that pure substances with an A.I.T. in the neighbourhood of 557° C. have the highest anti-knock value.

The benzene-toluene-xylene series have A.I.T.'s of 656°, 633°, and 618° C. and their H.U.C.R.'s are in the reverse order.

This relation seems to hold for any given series of homologous compounds but cannot be used for substances which are unrelated in structure. The n-hydrocarbon series follows the reverse order (the A.I.T.'s being lower than 557° C.)

It is postulated that in any homologous series the A.I.T. and the anti-knock rating will pass through or move toward or away from a maximum or minimum as one moves up or down the series, also that the curve of A.I.T./number of carbon atoms will be parallel to the curve of H.U.C.R./ carbon atoms, further that for each series there will be a critical A.I.T. corresponding to maximum H.U.C.R., the H.U.C.R. decreasing as the A.I.T. moves either upwards or downwards from this temperature.

When two or more homologous series are present in a mixture it does not seem possible to predict the A.I.T. or the H.U.C.R. of the mixture.

Curves are given showing the relation between A.I.T. and the anti-knock rating of blends of straight run gasoline and lead tetra-ethyl. The effect of the lead is first to lower, and then to raise the A.I.T., the actual values depending on the nature of the ignition surface. When benzene is added to gasoline the A.I.T. curve rises rapidly and then more slowly with increased concentration of benzene when a quartz ignition surface is used, but with a platinum surface small additions of benzene cause a fall of A.I.T. followed by a rise after about 12 per cent. is reached.

Comparison of the A.I.T.'s of a number of straight run gasolines with their anti-knock ratings shows good relationship with a minimum value of A.I.T. of about 510° C. for the highest H.U.C.R. Cracked gasolines do not fall on as good a curve but there is still a general correlation possible. The minimum A.I.T. in this case is followed by a rise with further increase of H.U.C.R.

Tests made with lead-platinum, glass, platinum, and gold as the igniting surfaces show that the gasolines of low anti-knock value are most affected by the catalytic activity of the surfaces.

When lead tetra-ethyl was added to pure liquids of different A.I.T. the general effect was to raise the A.I.T.'s of the lower A.I.T. liquids and to lower the A.I.T.'s of the higher A.I.T. substances towards an optimum value. The effect of carbon disulphide is also to change the A.I.T. towards an optimum temperature.

It is pointed out that the air-fuel ratio undergoing initial ignition in the apparatus is not known definitely, but it is thought that the ignition temperature measured is that of the air-fuel ratio having the minimum ignition temperature. R. S.

Cracking.

510. Flue Gas Recirculating System for Cracking Stills. H. A. Atwater. U.S.P. 1,709,764. April 16, 1929. Appl. May 9, 1927.

The invention relates mainly to the maintenance of proper heat conditions at the heating surfaces of cracking stills. It is intended to provide a more efficient and economical system whereby uniform temperature and practical control may be possible by recirculating a portion of the flue gases through the combustion chamber. In the type of plant illustrated, a bank of tubes spanning the upper part of a combustion chamber, forms the heating surface of the cracking unit. An extension of the lower part of the combustion chamber is built in three horizontal sections. The upper and lower sections have chequer walls at the inner end and serve to introduce the recirculated gases without over-chilling the furnace. The middle section is the fire box. The waste gases from the combustion chamber pass into a flue leading to a

stack, and from this flue a portion of the gases is drawn by a fan and returned to the fire box section. The quantity of gases returned may be controlled to produce the desired temperature in the combustion chamber. R. P.

511. Process of Cracking Oil. J. C. Black. U.S.P. 1,715,980, June 4, 1929. Appl., Oct. 20, 1926.

The process is designed to produce, by cracking a crude oil residue, a relatively small amount of gasoline, a larger amount of higher boiling oil, and a residuum suitable for fuel purposes. The crude residue is passed through a series of cracking coils under pressure at a temperature sufficient to crack the residue, and the heating is prolonged, but destructive decomposition to carbon is prevented by the introduction of a cool oil into the final stages of the last cracking coil, in quantities sufficient to maintain the crude oil below the temperature of coking and to lightly crack the cool oil introduced. The product from the coils is passed into a fractionating column and the gasoline, gas oil and residuum are separated in the usual manner. R. P.

512. Process for Converting Petroleum Oils. W. M. Cross. U.S.P. 1,717,007, June 11, 1929. Appl., Oct. 14, 1922.

The apparatus consists of the usual cracking coil, a reaction chamber and a condenser, but a tube is placed along the bottom of the reaction chamber for the purpose of introducing air or other combustion supporting gas. The process consists of heating the oil in the coil to a predetermined temperature, which depends on the oil to be treated. With Mid-Continent gas oil a temperature of from 700° to 950° F. is suitable. The highly heated oil is discharged from the coil into the reaction chamber, which is maintained at a pressure ranging from 400 to 900 lb. per sq. in., according to the oil used and the character of the product desired. The oil passes slowly through the reaction chamber and has sufficient time to be subjected to the cracking conditions and digestion therein. During the reaction there is introduced, through the tube placed in the reaction chamber for the purpose, a controlled quantity of air or oxygen. It is essential that the air or oxygen is charged only at such times as the temperature of the oil in the reaction chamber is above the flash-point of the oil. The extent of combustion and the heat evolved depends directly upon the quantity of gas introduced. Thus the temperature of the oil body may be accurately controlled. By controlling the temperature in the conversion chamber it is possible to reduce the temperature and lessen the risk of carbon deposition in the heating tubes. Also there will be an agitation in the reaction chamber which will prevent the deposition of carbon in the chamber and lengthen the running time of the plant. R. P.

513. Process for Treating Oil. R. J. Dearborn. U.S.P. 1,658,077, Feb. 7, 1928. Appl., April 18, 1922.

This invention relates to the conversion of high-boiling hydrocarbon oils into low-boiling hydrocarbon oils, and has special reference to a cracking process for producing gasoline in which aluminium chloride is employed as a catalytic agent. Heat is applied to the oil which is completely free from aluminium chloride, the oil then being discharged into a vaporising or distilling chamber, where it is brought into contact with the catalyst without any additional heat being supplied. By this means the oil is raised to a temperature sufficient to enable it to crack in the presence of the aluminium chloride, with the production of low-boiling hydrocarbon vapours. A portion of the heavier components of the vapours is condensed and mixed with the incoming oil in order to be passed through the heating zone, and then returned to the reaction zone. The remainder of the vapours is condensed to gasoline.

H. E. T.

514. Process for the Conversion of Hydrocarbon Oils. J. F. Donnelly. U.S.P. 1,712,789, May 14, 1929. Appl., Nov. 21, 1924.

The process consists of heating a mass of oil to a cracking temperature under pressure sufficient to prevent vaporisation and discharging the heated oil, before the cracking reaction reaches the stage where free carbon is formed, into a low-pressure zone containing a separate mass of hydrocarbon oil, thus checking the cracking reaction and preventing the formation of carbon in the low-pressure zone by the regulated intermixture of a cooler oil. The vaporised portion of such intermixture is subjected to fractional condensation, and a regulated portion of the condensate is returned to the mass of cooler oil in order to maintain it at a temperature which will prevent the formation of free carbon.

The apparatus comprises a pipe still, in which the oil is raised to cracking temperature. The heated oil passes through a nozzle into a long chamber situated centrally within a large horizontal still, and a series of outlets extend from the central chamber to a point near the bottom of the still. From the bottom of the still a conduit leads to the space round the nozzle, thus the high velocity of the oil leaving the nozzle draws up the residue from the bottom of the still and mixes it with the stream of expanding oil or vapour issuing from the nozzle.

A condensing tower is mounted on the still, and the condensate from this column flows into a secondary still having a vapour outlet back to the tower. A vapour line leads from the top of the tower to a water condenser, thence to the gasoline receiver. The secondary still is also provided with a fresh oil supply and a constant level is maintained therein. From this still the feed pumps draw the charge to be treated in the pipe still. R. P.

515. Process for Treating Oil. C. P. Dubbs. U.S.P. 1,716,306, June 4, 1929. Appl., Oct. 6, 1920.

A series of pressure stills are connected so that the oil fed into the first of the series may flow successively through each still of the series. A dephlegmator is attached to each still and connections are arranged to pass the reflux condensate into the next still without permitting any of the condensate to return to the still from which it is derived. Each still is set in a separate heating zone, and the temperature of the oil is increased as it passes through the series. Thus the condensed oil from the first dephlegmator flows to the second still to be treated at a higher temperature, and the condensate from the second dephlegmator flows to the third still to be again treated at a still higher temperature, and so through the process. The mixture of vapour and gas from all the dephlegmators is cooled and the gasoline separated from the permanent gases by the usual methods. R. P.

516. Process for Cracking Oil. G. Egloff and H. P. Benner. U.S.P. 1,659,398, Feb. 14, 1928. Appl., Jan. 11, 1926

This invention is a continuation of the previous application Serial No. 445,819, and relates to improvements in the cracking of petroleum oil by heat and pressure. The speed of the cracking reaction, as well as the character of the reaction, is facilitated by increasing the speed of hydrocarbon vapour as it passes through the expansion chamber, thus causing a substantial increase in frictional heat. This is accomplished in the apparatus by gradually decreasing the cross-sectional area of the elongated vapour chamber. The active principle adopted is the slowing down of the liquid velocity as it passes into vapour in the expansion chamber, by causing it to pass into a vessel of

large dimensions and then gradually increasing the vapour velocity as it passes through the expansion chamber. As the hydrocarbon vapours pass through the constantly narrowing expansion chamber, frictional heat is generated, which increases the rate of reaction.

A typical run in the apparatus shown in the diagrams would consist in charging a 24 Baumé gravity fuel oil through heating tubes at the rate of 25 barrels per hour, the amount of oil being relative to the heating area, the velocity with which the vapours in the expansion chamber are increased, and also to the dephlegmating area. By operating the plant at a pressure of 150 lb per sq. in., and a liquid transfer temperature from the tubes of 825° F., 25 barrels of oil will be converted into 7·5 barrels of 58° Baumé gravity gasoline per operating hour. H. E. T.

517. Submerged Combustion Process. C. Ellis, Assr. to Ellis-Foster Co. U.S.P. 1,716,433, June 11, 1929. Appl., March 17, 1925.

Gas oil or other material to be cracked is placed in a horizontal or vertical still, and into the oil is forced a preheated inflammable oxygen-containing mixture capable when ignited of burning beneath the surface of the oil, yielding a submerged flame. The burner preferably depends from the top of the still into the oil, so that the combustible mixture is forced downwardly in the oil, subsequently rising to be carried away along with the products of cracking and distillation. A pressure above atmospheric is preferably maintained in the still. H. S. G.

518. Apparatus for Treating Hydrocarbons. L. de Florez. U.S.P. 1,715,643, June 4, 1929. Appl., July 8, 1919.

The apparatus has been designed for the cracking of oil in the vapour phase In order to reduce the risk of coking the heating coil is composed of tubes gradually decreasing in diameter, arranged in such a manner that the cold oil is heated in tubes of large cross-sectional area, and as the temperature increases the diameter of the heating tubes decreases, thus enormously increasing the velocity of the oil stream. From the heating coil the vapours pass into an expansion chamber, thence to a carbon settling drum, and finally through a condenser and separator where the permanent gas is separated from the gasoline. It has been observed that, if during the cracking operation, a portion of the gas be withdrawn from the system, an additional amount of gaseous products will be formed, thus increasing the gas yield at the expense of the liquid product. To avoid this loss a portion of the gas produced is injected along with the raw oil, so that an equilibrium between the gaseous and liquid products will be established to minimise the formation of new gas. It is contemplated that the formation of less gas for each increment of raw material will tend to increase the yield of liquid products. The gas may be injected at different points of the process, as desired. R. P.

519. Industrial Process for the Production of Liquid Hydrocarbons by Cracking and Simultaneous Hydrogenation. J. Fohlen. *Chimie et Ind. XXI*, 6, 177–84.

The principle of this process depends on the decomposition of organic molecules into the nascent state resulting from the cracking of carbonaceous matter, and hydrogenation brought about by the action of atomic hydrogen. The process is carried out at almost normal pressure, and the atomic hydrogen is prepared by electrical, chemical or catalytic means.

The plant in operation at "Laboratoire des Mines de la Sarre" is described in detail and various diagrams shown. The operation is effected in two

phases (1) by cracking without condensation, (2) by catalytic hydrogenation without condensation but with intermediate desulphuration.

The initial substances (oil, lignite, schists, tar, etc.) are cracked, using metallic chlorides as catalysts. After cracking without intermediate condensation the gases and hydrogenated vapours are passed over the refining elements, which consist of the same types of metals as the catalysts—*i.e.*, nickel, copper and iron—maintained at the reaction temperature by the vapours of the hot gases in the distillation retorts. The refiners consist of two groups of identical elements, one group which refines and the other group is regenerated by roasting in a current of air or oxygen. The vapours (C_4H_4S H_2S, etc.) react on the metals of the refiners, forming sulphurated and arseniated elements which react on the refining metals to produce sulphates, arseniates, and hydrogen or nascent organic radicles, and these take part in the reaction with the other unstable tar substances. Atomic hydrogen reduces the sulphate to sulphur. The hydrogenated gases and vapours pass, immediately after refining, without intermediate condensation into the catalytic tubes at a temperature of about 300—350°. These tubes contain, like the desulphurisers, nickel. Phenols are removed here. The spirit is recovered by absorbing it in active carbon.

The results obtained by the "Société d'Etudes et d'Exploitation des Matières Organiques" are given. W. S. C.

520. Specification Fuel Oil from Heavy Cracked Residue. R. T. Goodwin. *Nat. Petr. News*, May 15, 1929, XXI, 26, p. 80.

The Goodwin process for the stabilization of cracked fuel oil consists of chemical and mechanical treatment to produce a merchantable bunker fuel and a carbonaceous residue suitable as a refinery fuel or a briquette binder.

The residuum after leaving the cracking plant contains two sedimentary materials; one of these results from the lime used, which forms calcium sulphide, and the other is carbonaceous matter resulting from the presence of unstable high carbon hydrocarbons. The process consists in treating the residue from the cracking plant, after cooling to about 275° F., with spent acid from the refinery, first in a Dorr flocculator, then in a Dorr tray thickener. The oil from the thickener is clean and stable, and the slurry, about 10 per cent., is filtered, yielding a further 5 per cent. of oil and 5 per cent. of filter cake. The clarified oil is suitable to market specification as a bunker C fuel.

The Dorr tray thickener is a mechanically operated continuous settling machine. It consists of a cylindrical tank divided into several superimposed compartments by a series of dish-shaped steel trays attached to the sides of the tank. A slowly revolving central steel shaft is provided with radial arms having plough blades attached. One set of arms in each compartment sweeps the settled solids to the centre, where they drop to the bottom of the tank.

The treated residuum enters through a well at the top, the settled solids are removed from the bottom by a pump, whilst the stabilized finished oil overflows from connections at the top of each compartment.

The filter cake contains approximately 85 per cent. of carbon and has a B.Th.U. value of 15,000 to 18,000. It may be mixed and burned locally with coal, or it may be briquetted with anthracite screenings. Briquettes require a comparative low temperature of roasting, about 500—600° F. The oils experimented upon contained 5 to 25 lb carbon per barrel, depending on the type of cracking plant used, but the process is particularly useful where lime has been added to the raw cracking stock to neutralise the corrosive effect of sulphur, and in cases where the residue continues to deposit asphaltic substances over a long period. R. P.

521. Art of Cracking Hydrocarbons. E. C. Herthel and H. L. Pelzer. U.S.P. 1,714,090 and 1,714,091, May 21, 1929. Appl., June 11, 1927.

A charge of oil is circulated from a large pressure still through a battery of heating tubes back to the still. A body of finely divided filtering or desulphurising material is supported above the bottom of the still. The oil is drawn from beneath this material, and after passing through the heating tubes is discharged above it. The material may be fuller's earth, 15—30 mesh, or pulverised coke, supported on a wire gauze screen dividing the still.

The temperature in the still is maintained in two ways, indirectly by heating the circulating oil in the tube heater and directly by passing heated cracked gas into the oil beneath the filtering screen. This gas is heated in a separate furnace and heating coil. In this way the burden of heat transfer through the tubes is reduced, with a corresponding reduction in the tendency to form carbon deposits. At the same time, these tubes are protected by circulating the oil through the bed of filtering material to reduce the pitch or asphaltic constituents. Further, the supply of raw oil and the removal of cracked residue are regulated to maintain the concentration of these products in the bulk of the oil below saturation, or below an objectionable value.

The gaseous heating medium introduced in the manner indicated serves to maintain part of the finely divided material in suspension in the oil, thus promoting intimate contact with still charge and maintaining free circulation of the oil through the screen.

In a pressure still in which the normal charge is 10,000 gallons, 4 to 7 tons of fuller's earth or other clay is used, and to make up for the material carried away by the residue during operation additions may be made in suspension in the raw oil.

Pressure varies, but with gas oil a pressure of 125 lb. per sq. in. may be used. The vapours from the still pass through a reflux tower to the condenser and receiver. The reflux oil returns into the suction of the circulating pump, to be cracked again with fresh oil. R. P.

522. Process for Producing Liquid Hydrocarbons. F. J. M. Hansen. E.P. 284,224, April 24, 1929. Appl., Jan. 24, 1928.

This invention relates to a process for producing liquid hydrocarbons by hydrogenation, wherein the material is in contact with hydrogen at a place where the latter is caused to dissociate into the atomic state. The process is based on the discovery of the inventor that the dissociation of hydrogen is inhibited by the presence of foreign matter such as gaseous or vaporised hydrocarbons, but pure hydrogen is easily dissociated. Accordingly, the hydrocarbon is introduced into the electric zone only after the dissociation of the hydrogen has started. As the dissociation slackens after the introduction of the hydrocarbon, it is advantageous for continuous working to interrupt periodically the introduction of the hydrocarbon into the zone of dissociation, hydrogen alone being supplied during the interruptions. For example, naphthalene may be hydrogenated as follows:—A current of hydrogen is passed through a suitable series of electric arcs in which it becomes dissociated in known manner. After dissociation has started impure naphthalene vapour is introduced into the arc together with hydrogen. The atomic hydrogen produced attaches itself easily at low pressures to the naphthalene. Care must be taken that the product is very rapidly removed, otherwise the hydrogenation product will be decomposed, and it is advantageous to interrupt the introduction of the naphthalene periodically for the reasons already stated. R. P.

523. APPARATUS FOR TREATING OILS. W. R. Howard. U.S.P. 1,715,066, May 28, 1929. Orig. Appl., Sept. 13, 1922. Divided March 31, 1924. Renewed June 2, 1927.

The raw oil is pumped into a heating coil and raised to a cracking temperature; thence it flows into a pan suspended in an enlarged expansion chamber. This pan is preferably of such size proportionate to the size of the expansion chamber that it will have a capacity to hold the heated oil introduced from the coil for a period of time sufficient to allow maximum vaporisation. The vapours are drawn off to a dephlegmator where part of the vapours condense, whilst the uncondensed gas and vapours pass overhead to a condenser and receiver. The condensate formed in the dephlegmator returns to the expansion chamber but falls below the pan, and a distillate residue may be drawn off from the bottom of the chamber. It is desirable that this be kept separate and not mixed with the raw oil.

Another draw-off line is connected to the bottom of the pan in the expansion chamber. This residue draw-off line is connected to a still which is heated by a burner, and steam may be introduced from an extraneous source. The still is provided with a condenser, a receiver, and a residue run-off pipe, either running to a receiver or back into the heating coil for further treatment of the oil.

The expansion chamber may be maintained at the same temperature as the heating coil, or it may be raised to a higher temperature by means of additional burners. The entire system is maintained at a uniform pressure developed by the generated vapours, and is controlled by a valve on the outlet of the receiver.

The residue from the pan is run to the still and the pressure reduced to atmospheric. A large part of this residue is vaporised, and the portion which does not vaporise by the latent heat, aided by the reduction in pressure subjected to the agitating action of steam and distilled, if desirable to coke or drawn off and returned to the heating coil for further treatment. R. P.

524. TREATMENT OF SOLID CARBONACEOUS MATERIAL FOR THE RECOVERY OF OILS. W. A. Humphrey and Imperial Chemical Industries, Ltd. E.P. 305,744, Feb. 14, 1929.

Solid carbonaceous material (*e.g.*, coal), which may suitably be crushed to particles of less than ¼ in. diameter, is distilled in the presence of oil in such a way that the solid in moving through a continuous body of the oil is subjected to progressively increasing temperatures. The range of temperatures advantageously extends from the lowest at which volatile matter is disengaged from the carbonaceous material to the highest permitted by the b.p. of the oil under the conditions of working. The process may be conducted in a body of oil contained in a vertical tower, the solid material being added at the top where the temperature is lowest and allowed to sink through the oil, thus encountering progressively increasing temperatures, residual solid material and oil being removed at the base, and gases and vapours being withdrawn from the top. The tower may be provided with baffles to retard the downward movement of solid particles. Methodical transit of the solid may be insured by causing the body of oil to surge up and down periodically; thus, a U-shaped tower may be employed and means provided for alternately releasing and building up pressure at the head of each column, or a reciprocating piston at the base of the U may be employed. Steam or hydrogen or other gas (which may be pre-heated) may be passed up the tower and the effluent gases treated for recovery of oil and then recirculated, possibly after enrichment with hydrogen or steam. The process may be carried out under

pressures ranging from atmospheric to those of the order used in the destructive hydrogenation of carbonaceous material (*i.e.*, 100 atm. or more). Settling of the residual solids, preliminary to separation by centrifuging or filtration, may be effected externally to the tower or in a lower portion of the tower below the steam and/or gas inlets. W. S. N.

525. Destructive Hydrogenation of Carbonaceous Material. H. A. Humphrey and Imperial Chemical Industries, Ltd. E.P. 308,995, April 2, 1929.

The destructive hydrogenation of solid carbonaceous material—*e.g.*, coal—is carried out in suspension in such an excess of liquid medium of high b.p. that the solid may move freely, a vertical tower (*i.e.*, a vessel very high in proportion to its width) being used, and hydrogen or gases containing hydrogen being employed under a pressure of about 100 atm. or more. Suitable temperatures are 300—550°. The liquid medium is an oil of simple chemical composition not too readily decomposed under the conditions employed—*e.g.*, oil from a previous hydrogenation. The hydrogen, which may advantageously be preheated, is introduced at the base of the tower. The solid carbonaceous material may be introduced at the top and allowed to descend by gravity, residual solids (unconverted material, ash, catalyst) being removed at the base. Alternatively, the solid may be introduced at the base and the additional oil formed withdrawn from the top of the tower, *cf.*, E.P. 305,744. W. S. N.

526. Manufacture and Production of Valuable Liquid Hydrocarbons. J. Y. Johnson. From I. G. Farbenind. A.-G. E.P. 311,251, Dec. 5, 1927.

Coal, tars, mineral oils, etc., are destructively hydrogenated or cracked in the presence of catalysts consisting of metals or their compounds or both, if desired, deposited on carriers suitable for the process which have not been employed in a previous cracking stage, which have been subjected to a preliminary treatment at an elevated temperature with gases such as carbon dioxide, sulphur dioxide, nitrogen oxides, oxygen, air, nitrogen and the like, which have no reducing action upon them. H. S. G.

527. Conversion of Hydrocarbons of High B.P. into those of Low B.P. J. Y. Johnson and I. G. Farbenindustrie Aktiengesellschaft. E.P. 311,789, May 14, 1929.

"Ansolvo" acids (*Annalen*, **455**, pp. 235, 244 and 249) are acid complexes obtained by mixing an organic acid with certain types of inorganic salts. Such complexes have a greater power of giving off hydrogen ions than the organic acids from which they are derived. Thus, an "ansolvo" acid causes the liberation of nitrogen from diazoacetic acid or ester more readily than does the parent organic acid.

It is now proposed to employ "ansolvo" acids, or substances which give "ansolvo" acids by interaction, as catalysts in the thermal decomposition or destructive hydrogenation of coal, oil, tar, etc., the process being carried out at 350—600° (preferably at 400—450°) and at pressures from atmospheric up to 1000 atm., 100—150 atm. being very favourable. The operation may be conducted in the presence of hydrogen or of gases containing or supplying the same, or in the presence of inert gases such as nitrogen. Thus, 100 parts of a thin tar fraction b.p. 200—350° are treated with 5 parts of a double compound of α-naphthoic acid and tin tetrachloride at 350—400° and about 10 atm. Of the conversion products, 41·2 per cent. has b.p. up to 200° (calc. on the initial material).

A rectified thick producer tar (100 parts) and 5 parts of a double compound of 1-naphtholcarboxylic acid and aluminium chloride, or the components of the same, are heated at about 380—420° in an autoclave lined with Monel metal. Of the resulting products, 33·5 parts have b.p. below 200°.

A thick producer tar (100 parts) is treated with 3 parts of a double compound of oleic acid and zinc chloride, or the components thereof at 350° and 25 atm. in a stirrer autoclave. Of the products, 35 parts have b.p. below 200°

Paraffin wax, m.p. 41—43° (100 parts) is heated with 1 part of a double compounds of formic acid and aluminium chloride, or the components of the same, at 400—440° and 30 atm. Ninety-nine parts distil over, of which 75 parts boil below 200° and a further 24 parts boil between 200° and 360°.

A mineral oil, b.p. above 200° (100 parts) is heated with 5 parts of a double compound of naphthoic acid and antimony pentachloride, or the components thereof, under a pressure of 50 atm. of hydrogen, up to 420°. Of the resulting products, 45 parts have b.p. below 200.

It has already been proposed to carry out the distillation or cracking of carbonaceous material in the presence of a small quantity of the admixed carbonates of sodium, potassium or calcium, formic acid vapour being admitted into the distillation or cracking vessel. In this process, however, "ansolvo" acids are not obtained. W. S. N.

528. Motor Fuels and Other Products from the Cracking of Wood Tars. J. C. Morrell and G. Egloff. *Ind. Eng. Chem.*, Vol. XXI, **6**, 537–542.

The possibilities of the utilisation of waste wood products, particularly tars derived from the destructive distillation of wood, by conversion into motor fuels and other products by the cracking process, are pointed out by means of data obtained by cracking three types of wood tar. As an example of the possibilities of the utilisation of the annual accumulation of waste wood may be cited the fact that from an annual yield of 7·5 billion board feet of Douglas fir, or 4·2 million tons, the following products are obtained :—

Turpentine and light oils	8,500,000 gallons
Tar	70,000,000 „
Wood alcohol	9,780,000 „
Acetate of Lime	187,500,000 pounds
Charcoal	2,440,000,000 „

while a large yield of gas for fuel is also obtained. The 70,000,000 gallons of tar will, according to this research, produce by cracking 23,300,000 gallons of motor spirit, equal in anti-knock value to benzene. In addition, approximately, 6,000,000 gallons of low boiling tar acids or phenols for antiseptic, disinfectant or other purposes are produced. Further, 125,000 tons of high grade coke, and 1,830,000,000 cubic feet of gas will be made during the cracking process.

A wood tar derived from the thermal decomposition of Douglas fir was subjected to the cracking process under pressures of 75, 100 and 120 lb per square inch, and temperatures ranging from 393° C. to 405° C. The yield of tar acid free motor fuel produced was approximately 26 per cent. The anti-knock value of this fuel was better than that for the aromatic hydrocarbon benzene, while an engine test rated it as equivalent to 100 + per cent. benzene equivalent. Approximately 30 per cent. of the tar-acid-free motor fuel can be made from the gas oil produced during cracking by recycling, thus bringing the total yield of motor fuel up to 33 per cent.

The gum present in the motor fuel was determined by the U.S. Bureau of Mines method, and found to be 140 mgm. per 100 ccs. The tar acids in the unrefined motor fuel were about 19 per cent., and a finished motor fuel was

produced by washing the crude spirit with caustic soda. Analysis of the motor fuel showed that it contained 97·2 per cent. of aromatic hydrocarbons and 2·8 per cent. of paraffins and naphthenes.

The gas oil or pressure distillate bottoms make excellent recycling or blending stock for recracking.

Pine tar oils were next subjected to the cracking process, and the average yield of distillate boiling within the motor fuel range varied from 22 to 41 per cent. Depending on the product desired, the pressure distillate may be fractionated into motor fuel and gas oil or into solvent oil, turpentine substitute or fine oil equivalent. A composite sample of the above products after refining showed a gum value of 1,193 mgs. per 100 ccs., which is far too high to permit the use of this distillate as motor fuel. Hence the cracking of pine tars will be better directed towards the production of solvents than for the production of motor fuel. By cracking tar derived from hardwoods, it was concluded that, owing to the extremely high tar acid content, the production of motor fuel from this type of tar will not be profitable.

The refining of cracked distillates from wood tars is similar to the refining of cracked distillates from low temperature coal tar and from shale oils. The tar acids are first removed by successive washes with 20 to 25 per cent. solution of sodium hydroxide; the cracked distillate is then water washed and treated with dilute sulphuric acid. The treated oil is finally steam distilled over activated clay, in order to stabilise the fuel and lower its gum content. Finally the product is washed with soda to improve its odour and stabilised by treatment with fuller's earth. H. E. T.

529. Cracking Mineral Oil. A. E. Pew and H. Thomas. U.S.P. 1,692,786, Nov. 20, 1928. Appl. Jan. 25, 1926.

The feature of the process is the method adopted for heating the oil by means of mercury vapour. In usual methods of heating oil to cracking temperature, the difference between furnace gases and oil is very great. This causes carbon formation in the film of oil in contact with the tube, but by using mercury vapour as the heat carrier the difference in temperature between oil and vapour is relatively small. The plant consists of a mercury boiler situated within a furnace, and a preheating coil between furnace and smoke stack. The cracking units consist of a coil of tubing within a casing. The oil to be cracked is pumped through the preheater, then successively through the cracking units, of which there may be several. The mercury vapours from the boiler surround the cracking tubes and condense, giving up their latent heat. No hard or insoluble carbon is formed in the tubes, but there is a deposit of asphaltene which may be removed by solution with carbon disulphide. R. P.

530. Manufacture of Synthetic Liquid Fuels. E. A. Prudhomme, Assr. to Soc. Internationale des Procedes Prudhomme (S.I.P.P.), Paris. U.S.P. 1,711,856, May 7, 1929. Appl. April 7, 1926. Fr. March 9, 1926.

Hydrocarbon products are vaporised and passed through a dephlegmator maintained at 400° C. to separate out heavier fractions, and the volatile products passed through a purifying element maintained at 350° C. They are then subjected to the action of a plurality of catalysers effecting in successive stages a general hydrogenation, a splitting up of hydrogenated products into unsaturated hydrocarbons, and a saturation and polymerisation of the unsaturated hydrocarbons, while maintaining a temperature of not more than 250° C. throughout the catalysers, and adding to the products a separate quantity of reducing gas in each catalyser. The resultant product is passed to a suitable condensing apparatus. H. S. G.

531. Oil Cracking Apparatus and Process. A. Schwarz. U.S.P. 1,714,453 and 1,714,454, May 21, 1929. Appl. March 25, 1925.

In cracking processes commonly used for the production of motor fuel it is more or less common practice to crack the oils in such a way that coke and heavy tars of little value are produced. Furthermore, the heat transfer is usually through the walls of the containers of the oil, which is wasteful and frequently causes the deposition of carbon on the heating surfaces. In this process practically all the heat is supplied directly to the oil without passing through the walls of the receptacle. Another object of the invention is to consume all the residue formed to generate heat and gas in order to eliminate the handling of residues by producing gas and spirit only.

The plant consists of a gas producer similar to those commonly used but modified to provide an upper still portion. Coal or coke is fed into the producer and combustion started by blowing air through the charge. When the temperature near the top of the coal or coke reaches 700 to 1,000° F. a vacuum pump at the outlet of the apparatus is started and draws air through the incandescent coke and the remainder of the apparatus. Superheated steam is fed from below the mass of fuel, and at the same time oil is injected into the coal or coke where cracking takes place. The vapours pass downwardly and carbon is deposited, then the mixture of gaseous products from oil and coal pass through a conduit to a settling chamber, where suspended particles are arrested, thence to the fractionating column. Spirit and gas pass overhead to a condenser and separator, and the reflux residue may be returned through the system. If air is continuously admitted fatty acids are produced which may be used for many purposes, but for the production of motor fuel air is excluded. R. P.

532. Cracking of Hydrocarbon Oil. Standard Oil Development Co. E.P. 311,362, May 10, 1929. Appl. date, Feb. 10, 1928.

In this process a cracking coil mounted within a furnace raises the temperature of the oil to be cracked to over 800° F. The oil is discharged into a vertical digestion drum capable of withstanding a pressure of at least 300 lb per sq. in. The coil extends into the centre drum at a point about one-third of the height and discharges in an upward direction. The oil level is maintained at a considerable height above the coil discharge.

Vapour evolved from the liquid passes into a second vertical drum fitted with baffles in its lower section, and a preheating coil is placed at the top of this tower to preheat the raw feed and provide reflux. The reflux oil is returned to the heating coil at a point near the discharge into the digestion drum. Liquid from the bottom of the digestion drum is withdrawn and discharged in intimate contact with vapours from the tower into a separator, also in the form of a vertical drum.

In this separator a substantial reduction of pressure takes place. The separator is provided with fractionation plates, a steam spray at the bottom line for the removal of unvaporised tar, and a cooling coil is placed at the top of the separator. The condensate produced is caught in a pan above the tar space and removed separately, whilst the uncondensed vapours pass overhead to a water condenser. Thus the products consists of gas, gasoline, an intermediate oil and a heavy tar. R. P.

533. Cracking and Rectifying Petroleum Oils. J. M. Wadsworth. U.S.P 1,692,476, Nov. 20, 1928. Appl. March 12, 1926.

The cracked oil from a reaction chamber is allowed to pass into the middle section of a column under a regulated pressure. The residuum flows from the bottom of the column through a heat exchanger to preheat the incoming

crude stock. Recycle oil is drawn from the middle of the column, and the vapours pass overhead to a heat exchanger through which the cold raw feed is forced on its way to the residuum heat exchanger. The vapours still under pressure pass into the middle part of a second tower, from which dry gas flows at the top and rectified gasoline at the bottom. The dry gas is allowed to expand through an engine to supply power, and finally the gas, after expansion, is used as a cooling agent. By this method the gasoline and lighter hydrocarbons are separated from the recycle and residuum oils, and subsequently the permanent gas is separated from the rectified gasoline. The pressure resulting from the cracking is utilised throughout the rectification. R. P.

534. Destructive Hydrogenation of Carbonaceous Materials. H. G. Watts and Imperial Chemical Industries, Ltd. E.P. 311,197, Jan. 30, 1928.

In order to effect ready separation of the liquid reaction products of the destructive hydrogenation of carbonaceous materials from the remaining solid matter, a proportion of finely divided coal or coke, equal to 10—50 per cent. of the amount of solid matter in the liquid hydrogenation product, is added together with a diluent oil, if desired, before subjection to filtration, preferably through a rotary filter whilst still hot. The filter coke remains as a fuel undiminished in value by the addition of the filtering aid. H. S. G.

535. Destructive Hydrogenation of Carbonaceous Materials. H. G. Watts and Imperial Chemical Industries, Ltd. E.P. 311,198, Jan. 28, 1928.

The crude reaction product from the destructive hydrogenation of carbonaceous materials is freed from constituents of b.p. up to 190° C., and the product diluted with 10—50 per cent. of an oil capable of dissolving pitch and preferably an oil of relatively high b.p. derived from the hydrogenation process itself. Finely divided coal or coke may be added to aid filtration. After filtration the solid matter is washed with light oil and the residue may be briquetted and used as a fuel. The purified liquid product, together with the washings, is returned to the hydrogenation process, either as such or in the form of a paste with fresh coal. H. S. G.

536. Continuous Distillation of Hydrocarbons with Simultaneous Cracking of High-Boiling into Low-Boiling Hydrocarbons. H. Wolf, Assr. to Carburde Akt., Switzerland. U.S.P. 1,711, 869, May 7, 1929. Appl. Sept. 24, 1925. Ger., Nov. 3, 1924.

A middle oil is cracked by heating in a coil system under pressure or by passage through a heated metal bath or by means of a catalytic process, and immediately after issuing from the cracking apparatus and before a separation of the liquid and gaseous phase is possible the cracked product is expanded freely below oil contained in a vessel. It is preferable to employ a Körting circulating nozzle and to have the vessel filled with Raschig rings of similar devices. The amount of cooling oil and its temperature is so selected in relation to the amount of cracked product that a sudden cooling to temperatures which exclude any after reaction is effected. The cracked product absorbed by the cooling oil can be recovered by distillation in a crude oil distillation plant linked together with the cracking plant, by which means a simultaneous distillation is effected. H. S. G.

Oil-Field Technology.

537. The Use of Hydraulic Cement in Oil Wells. J. B. Case. *Petr. Times*, 1929, Vol. 22, 17–18.

The cementing of wells to prevent infiltration of water into the oil-bearing sands and dissipation of oil and gas into formations of lower pressure is discussed. The exclusion of water within an oil zone from the producing sands is generally more difficult than the stopping of top water, since the placing of the cement to arrest the water at its exact source, without restricting production is not easy; but the stratigraphic uniformity of exclusion and control of the water within the oil-bearing horizon is as important as shutting off the top water.

Low-pressure sands occurring in thick oil zones also present two problems: (1) their location, and (2) their segregation from high-pressure sands without loss of production. The latter involves co-operation between the operators in a field, when the principle of "stage development" can be brought into operation, and production can be secured from low and high-pressure zones separately rather than collectively. D. G. P.

538. Important Improvement in Gas Lift. R. Van A. Mills. *Oil & Gas J.*, 1929, **27** (42), 47, 93.

A recent development in the use of gas-lift is the use of a low-pressure "kick-off" method for deep wells of high fluid level, in the Greater Seminola field. These wells, which are over 4,000 ft. in depth, with fluid levels as high as 3,600 ft., usually take up to 24 hours to start flowing by gas-lift with pressures of 700 to 1,200 lb. By the low-pressure method, full flow is obtained in from 40 to 75 minutes with pressures of 300 to 460 lb.

Oil and gas are pumped into the pressure string, the oil tending to counterbalance the oil in the flow string. The gas pressure and proportion are increased as the operation proceeds, until only gas is injected. The gas is carried downward, being further compressed by the weight of the oil, until it is liberated into the base of the flow column, when it rises and expands, aerating the flow column. At first the fluid head in the pressure column exceeds that of the flow column, but the relative heads change until at the final flowing stage the pressure column is all gas and much lighter than the aerated flow column. The usual low-pressure "kick-off" takes 50 bbls. of oil and about 30,000 to 50,000 cu. ft. of gas, the oil being recovered before normal flow commences.

The only addition to ordinary gas-lift plant is one high-pressure oil pump for each lease, with a header and manifold to serve the wells as required. The method is simple, low in cost, and easy in operation, and is one of the most important advances in gas-lift operation. G. S.

539. Water Coning Prevention and Control. R. Van A. Mills. *Oil & Gas J.*, 1929, **27** (42), 50, 250, 252, 254.

To combat water coning it is advisable to restrict the rate of flow and to apply back pressure. Some successful results have been obtained by completely shutting-in the well, and allowing the fluids to readjust themselves, and it is believed that many wells can be saved by plugging before abandonment on account of water.

Water coning can best be prevented by applying flow and pressure control. After the trouble has started the best procedure is bottom hole plugging, pinching in the wells, or temporarily plugging the oil sand with cement to allow re-adjustment of the fluids. The most effective method of water control is the maintenance of gas pressure above a water cone in a producing formation. G. S.

540. New Inhibitor Discovered for Protective Pipe Coatings. H. K. Ihrig. *Oil & Gas J.*, 1929, **27** (42), 266, 268.

An ideal inhibitor for incorporation in a bituminous pipe coating should be soluble in bitumen, insoluble in water, soluble in corrosive liquors, capable of incorporation in the primer, without chemical effect on the rest of the coating, unable to alter the mechanical properties of the bituminous coating, cheap, and should have marked inhibitive powers against corrosive soil liquors.

A new inhibitor having these properties has been discovered, known as "Inhiburine." It is a coal tar product. G. S.

541. Conditions in Operation of Gas Lift. R. H. Pence. *Oil & Gas J.*, 1929, **27** (50), 34, 172, 174.

In the use of the gas-lift the oil is raised by the expansion of the compressed gas, and the method of expansion is a matter of interest in studying gas-lift. In practice neither the adiabatic or the isothermal expansion exist, the process in an oil or gas well being intermediate between the two and known as exponential expansion. A dry gas well blowing to the atmosphere will approach the adiabatic type, while a well producing oil with a low oil-gas ratio approaches the isothermal. In the majority of cases the expansion is exponential. To apply these principles to working conditions the quantitative conditions adopted from Lucke's *Engineering Thermodynamics* are stated :—

V_1 = intial (smallest) vol. of gas in cu. ft.
V_2 = final (largest) " "
P_1 = initial (highest) pressure of gas in lb/sq. ft. abs.
P_2 = final (lowest) " " " "
W = work in ft. lbs.
K = a constant.
T_1 = Temp. in ° F. abs. for state (1).
T_2 = " " " (2).
R = the gas constant ($=P_1V_1/MT_1$).
M = weight.

Any given quantity of gas confined and not subject to extraordinary thermal changes will suffer regular pressure changes for each unit of volume change and a regular volume change for each unit of pressure change. If the gas or the thermal conditions are steady the volumes and pressures will have the relative for the same mass :—

$$P_1V_1^s = P_2V_2^s = K.$$

The exponents may have any value, usually between 1 and 1·5 in practice, the precise value depending on the substance, the thermal conditions of expansion or compression, and the vapour condition as to moisture and superheat.

Related quantities for exponential and adiabatic changes in gases and dry vapours are :—

1. P and V $\dfrac{P_1}{P_2} = \left(\dfrac{V_2}{V_1}\right)^s$

2. P and T $\dfrac{P_1}{P_2} = \left(\dfrac{T_1}{T_2}\right)^{\frac{s}{s-1}}$

3. V and T $\dfrac{V_1}{V_2} = \left(\dfrac{T_2}{T_1}\right)^{\frac{1}{s-1}}$

General exponential change :—

1. $$\frac{V_1}{V_2} = \left(\frac{P_2}{P_1}\right)^{\frac{1}{s}}$$

2. $$\frac{T_1}{T_2} = \left(\frac{P_1}{P_2}\right)^{\frac{s-1}{s}}$$

3. $$\frac{T_1}{T_2} = \left(\frac{V_2}{V_1}\right)^{s-1}$$

The general equations involving the item of work are as follows :—

For isothermal expansion $s=1$

$$W = K \log_e \frac{V_2}{V_1} = K \log_e \frac{P_1}{P_2}$$

where $K = P_1V_1 = P_2V_2$.

For adiabatic expansion the value of s is the ratio of the specific heats of the gas $= \frac{C_p}{C_v}$

For exponential expansion s may have any value other than 1 :—

$$W = \frac{P_2V_2}{s-1}\left[\left(\frac{V_2}{V_1}\right)^{s-1} - 1\right].$$

$$W = \frac{P_2V_2}{s-1}\left[\left(\frac{P_1}{P_2}\right)^{\frac{s-1}{s}} - 1\right]$$

$$W = \frac{P_1V_1}{s-1}\left[1 - \left(\frac{V_1}{V_2}\right)^{s-1}\right]$$

$$W = \frac{P_1V_1}{s-1}\left[1 - \left(\frac{P_2}{P_1}\right)^{\frac{s-1}{s}}\right]$$

The average value used for s is 1·25.

By Boyle's Law, if a volume of 1,000 cu. ft. of free gas is produced in a given time at a well head only 47 cu. ft. at 300 lb will be effective at the foot of the eductor. Thus :—

$$P_1V_1 = P_2V_2 \text{ or } 14{\cdot}7 \times 1000 = (300 + 14{\cdot}7) \times V_2$$

$$\therefore V_2 = \frac{14{,}700}{314{\cdot}7} = 46{\cdot}7 \text{ cu. ft. at 300 lb.}$$

When expansion has reduced the pressure to 200 lb the volume will be 68 cu. ft., and at 100 lb 128 cu. ft.

If the 1,000 cu. ft. of free gas has resulted from exponential expansion ($s=1{\cdot}25$) the effective amount at the foot of the eductor will be :—

$$\frac{V_1}{V_2} = \left(\frac{P_2}{P_1}\right)^{\frac{1}{s}} \text{ or } \frac{1000}{V_2} = \left(\frac{314{\cdot}7}{14{\cdot}7}\right)^{\frac{1}{1{\cdot}25}} \therefore V_2 = 86{\cdot}2 \text{ cu. ft.}$$

It is apparent that with isothermal expansion less gas is needed for a given amount of work.

The solubility of gases in liquids is familiar. Dissolved gases decrease the viscosity of the oil, influence the surface tension, and promote ease of flow. The average solubility of gas in oils was found to be $P = 4{\cdot}91V + 3{\cdot}01$. The average cubic feet of free gas per cubic foot of oil at gauge pressure $P = (\cdot 03625\ P) - \cdot 109$. The average solubility of air in cu. ft. per barrel is $P = 19{\cdot}6V + 16{\cdot}7$.

Gas is much more soluble in oil than air and has the added advantage that it decreases viscosity. G. S.

542. Effects of Speed on Pump Operations. R. R. Robinson. *Oil & Gas J.*, 1929, Vol. 28, pp. 41 and 153.

A dynamometer has recently been developed by the Martin-Loomis Corp. for taking indicator cards from a well pump, showing the exact polished rod load at all portions of the stroke. From a study of indicator cards taken from different wells it was found that a too fast stroke is detrimental in three ways: (1) volumetric efficiency is reduced; (2) range and value of rod stresses are increased; and (3) power consumption is greatly increased. A too low speed may reduce volumetric efficiency through lost stroke, due to rod stretch and slippage of the plunger. Each well has to be considered independently, since mechanical troubles, such as delayed valve action, gas lock, viscosity of fluid, and counterbalance partly affect the most economical speed. The best speed is determined in each case by a study of load changes occurring during the stroke. Formulæ for calculating the results obtained from the dynamometer are given. D. G. P.

Geology.

543. Genetic Connection between Salt Deposits and Petroleum. *Petr. Times*, 1929, 21, 906.

In *Inter. Zeit. f. Bohrtechnik, Erdolbergbau u. Geologie* (May 1) A. Weinfeld contributes an article on "New Theories regarding the Formation of Petroleum, etc.," the introductory part of which is a discussion of the genetic connection between salt deposits and the petroleum in porous sands. The author is opposed to the idea of migration of oil from its mother rocks, and assumes that the greater part of the oil found in porous beds is primary in character. He states that it has been established that all oil-bearing formations are productive of commercial quantities of oil only in the regions where ordinary salt occurs in quantity, and therefore it is logical to assume the genetic connection between the two substances. It is generally agreed that in the presence of much sodium chloride in solution in water, calcium sulphate cannot exist because it would be precipitated. (Haug: *Traite de Geologie*, p. 98, ed. 1911, is quoted in support of this.) The author therefore considers that this absence of calcium sulphate made it possible for soft bodied organisms (*e.g.*, without a calcareous shell) to exist in great abundance. These would decompose without leaving behind any fossil forms, and the remains would permeate and cover porous sands, thus forming the mother substance of petroleum. The water containing these organisms must necessarily be rather highly concentrated solutions of sodium chloride, otherwise the calcium sulphate in solution would give rise to other kinds of organisms with hard shelly exteriors. This is said to explain the genetic connection between petroleum and rock salt without the necessity of resorting to tectonic disturbances, fault lines or anticlines for the explanation. Even where such structures are in connection with salt deposits, they have on that account no direct connection with petroleum formation. Nevertheless, the anticlines are said by the author to have a certain influence in petroleum recovery, for they have brought the former salt lake bottoms, and with them the petroleum carrying beds, either to the surface or to within such distances below the surface that they can be economically exploited. The author claims that by this theory he arrives at a simple conception of the forming of primary petroleum without special regard to pressure, temperature or migration of the oil after formation. The theory naturally increases the range of formations in which oil may be expected to occur, as all formations which were subject to conditions more or less similar to those referred to above can be regarded as possible oil horizons. W. H. T.

Coal and Shale.

544. Low-Temperature Carbonisation: Modern Work on the Commercial Scale. I. The "K. S. G." Process. D. Brownlie. *Chem. and Ind.*, 1929, **48**, 569-572.

A large "K. S. G." (Kohlenscheidungs-Gesellschaft) plant of eight standard rotary retorts, each having a throughput of 65—100 tons per 24 hr., has recently been started up in New Jersey, U.S.A., and may be taken as typical.

Each retort consists essentially of two long, slightly inclined concentric drums, arranged to rotate slowly in a furnace. The inner drum is 85 ft. long and 5 ft. 6 in. diam., made of $1\frac{1}{32}$-in. steel plate, and carries the outer drum, 72 ft. long and 10 ft. diam., made from open hearth steel in three sections, the middle section being 1 in. thick, the end sections $\frac{7}{8}$ in. thick. The retort rotates once in 90 secs.

Small coal is fed to the lower end of the inner cylinder, which is provided with an internal spiral. This conveys the coal to the upper end in 1–1½ hr., the temperature rising to 200—300° C. The charge then falls through apertures into the outer cylinder, which at this end is the hottest zone (500—550° C.). Exhaust steam superheated to 450–500° C. is here blown through the charge to the extent of 5—10 per cent. of the weight of the charge. In the case of coking bituminous coal this sudden rise of temperature, combined with steaming, prevents the plastic material from expanding unduly. The low temperature carbonisation is then completed in another 1–1½ hr. at the other end of the outer cylinder, the temperature declining to 430—485° C.

Gases and vapours pass off from the upper end of the retort to a dust-extractor, after which they pass to the condensers and scrubbing towers, while the hot smokeless fuel leaves the lower end, being quenched only by cold gas, after which it is screened; 80 per cent. of the total is available for sale as domestic fuel and is claimed to be dense, hard, and free burning, in large pieces with 10–12 per cent. volatile matter. A certain amount of low-temperature fuel breeze (not over 10 per cent.) is blended with the raw coal to prevent the plastic charge from sticking to the retort walls.

The setting is heated by producer gas generated from the low-temperature fuel smalls. The gas is burnt in a combustion chamber and the resulting hot gases at 1300—1700° C. are diluted with 50 per cent. of the total combustion gases to give 600—700° C., passed round the retort in the absence of oxygen, and finally passed through the steam superheater.

The "K. S. G." process yields per ton of average 25–35 per cent. volatile coal:—

Gas (800 B.Th.U./c. ft.)	3500–5000 c. ft.
Crude light oil (total)	3 gal.
Tar (dry)	18–22 gal.
Smokeless fuel (10—12% volatile)	14 cwt.

The New Jersey plant includes a large water gas plant, so that a town gas of 535 B.Th.U./c. ft. can be produced by blending the rich low-temperature carbonisation gas with an equal volume of blue water gas of 300 B.Th.U./c. ft. The blended gases are freed from H_2S in purifying boxes. L. H. A.

545. Improvements in the Manufacture and Production of Valuable Liquid Products from Varieties of Coal, Tars, Mineral Oils and the Like. I.G. Farbenindustrie Aktien-Gesellschaft. E.P. 282,384. Apr. 9, 1929. Appl. Dec. 9, 1927.

This patent is an addition to specifications No. 249,155 and No. 277,273. When valuable hydrocarbons are produced from coal, tar, mineral oils and the like, at elevated temperatures and under pressures exceeding 50 atm.,

with gases containing hydrogen but free from substantial amounts of carbon monoxide and oxygen containing substances, it is sufficient to exclude injurious iron, nickel or copper from the reaction chamber. By injurious iron and nickel are meant such forms of these metals as are not physically or chemically resistant, and which initiate chemical reactions such as the formation of methane or coke, or polymerisation of the substances under treatment. These reactions may be induced by porous iron, or iron alloys containing impurities such as sulphur or oxidic slags, or even if the carbon content of the metal exceeds 0·5 to 1·0 per cent. Copper only produces injurious reactions if it contains sulphur.

The prevention of injurious reactions may be assisted by constructing those parts of the reaction chamber requiring protection, of alloys containing a high content of aluminium, chromium, molybdenum, tungsten, or vanadium. Electrolytically prepared coatings are not suitable for the purpose of this invention, as they are porous and are not sufficiently resistant. H. E. T.

546. Production of Valuable Liquid Products from Coal, Tar, Mineral Oil and the Like. Farbenind. Akt. I.G. E.P. 311,628. May 16, 1929. Appl., July 3, 1928.

The patent is a modification of the invention described in E.P. 33,339, and consists in constructing of silver or its alloys, or coating therewith, those parts of the apparatus which come into contact with sulphur-contaminated hydrocarbons at elevated temperatures. W. H. T.

547. The Influence of Mineral Constituents especially of Iron Oxide on the Hydration of Solid Fuels. B. Hlavica. *Brennstoff-Chem.* x, 10 (201-203).

The work described was performed to complete the earlier work on the hydration of coal in the presence of catalysts, in which it was found that ferric oxide, as well as oxides and chlorides of zinc, nickel, copper and cobalt were the best catalysts in this process.

10 per cent. ferric oxide when added to coal influences the hydration catalytically, and on the other hand it combines with the sulphur forming H_2S. In the present research the influence of ferric oxide on five types of coal (Karolina, Hedvika, Handlová, Prago and Jaroslav coals) was determined. Since naturally occurring solid fuels contain iron compounds, *e.g.*, as SO_4, pyritic sulphur, FeS_2, $FeCO_3$, and Fe in silicates, which themselves influence the organic masses, the coal ash and Fe present in the said forms in the coal, were determined.

Specially prepared dry, hard coal (produced by heating to 300° C. in an autoclave), of the following composition was utilised: Ash 1·60 per cent., carbon 87·53 per cent., hydrogen 3·62 per cent. and oxygen 8·55 per cent. referred to pure coal. Experiments were tried with and without catalysts. The iron content of the coal varied from 0·5–1·99 reckoned as Fe_2O_3. The remaining ash constituents, SiO_2, Al_2O_3, CaO, MgO, SO_3, etc. remained indifferent in the hydration. This was shown in the case of Karolina coals, to which Fe_2O_3 and 74·9 per cent. pumice were added when the oil product was not greater than 10 per cent. compared with the experiments without catalysts. This was only reckoned on Fe_2O_3 but FeS_2 appeared to have the greatest catalytic influence on the hydration.

Detailed results are given in tables of coal hydration without catalysts, and in the presence of ferric oxide. It was found that:—

(1) The coals hydrated without the addition of mineral constituents produced only small yields of tar.

(2) Naturally occurring coals always contain mineral constituents, especially iron sulphides which influenced the hydration catalytically whilst the total iron content in the coal was not so important.

(3) Fe_2O_3 acted as a catalyst during the hydration and gave a better yield of oils especially with older types of coal. The influence on younger coals and lignite was less.

(4) $ZnCl_2$ had a more intensive influence than Fe_2O_3 as shown in the case of coals with a higher carbon content. W. S. C.

548. Low-Temperature Carbonisation of Bituminous Material. J. Y. Johnson. From I.G. Farbenind. A.-G. E.P. 310,664. May 5, 1928.

In process of low-temperature carbonisation in which bituminous materials are passed from above downwards between internally heated heating bodies and walls of louvre pattern, the solid heating body is replaced by one consisting of a cooled skeleton frame supporting heating surfaces which are themselves subdivided into smaller units, thus allowing expansion without deformation and a higher degree of stability to be obtained. The heating surfaces should be constructed of material containing silicon carbide, or of pearlitic or electric cast iron, or of ceramic material of good heat-conducting capacity. H. S. G.

549. Working Down the Products Arising when Coal is Liquefied by Hydrogenation. K. Schoenemann. U.S.P. 1,709,957. Apr. 23, 1929

Solid substances (inorganic components of the coal and a certain amount of the coal itself), asphalt and liquid products from the berginisation of coal are separated by the addition of a relatively small amount of an aliphatic hydrocarbon oil, *e.g.*, gas oil. The proportion of the latter may be so chosen that precipitation of the asphalt does not occur until after the liquid portion of the mixture has been separated from the solids (*e.g.*, by decantation); a second separation (of liquid from precipitated asphalt) may then be carried out. The b.p. range of the diluent employed may be so chosen that it may be recovered when the solution, freed from solids and asphalt, is distilled.

Thus, 20 kgm. of raw petroleum are mixed at 200° with 100 kgm. of distillation residues having a b.p. (?) of over 280° and resulting from berginised pit-coal (softening-point, Krämer-Sandow, 35°, with 25 per cent. of insoluble matter). After about 1 hr., 40 kgm. of solid asphalt and insoluble components will have deposited. The supernatant oil is withdrawn. After a further 3–5 hr. another amount of 40 kgm. of soft asphalt free from insoluble compounds are precipitated and there remain 40 kgm. of a mobile, reddish-brown oil, low in asphalt content, which is separated as before. The precipitant is regained from all three fractions by steam-blowing. W. S. N.

550. Treating Low Temperature Tar and its Distillates. G. T. Morgan and D. D. Pratt. E.P. 307,382. Mar. 6, 1929.

Crude low temperature tar or a distillate from it is treated, at ordinary temperatures, with a solvent to precipitate the pitch. A distillate from low temperature tar, boiling up to 150° C., is preferred but ether or petrol may be used. The solution is settled or otherwise clarified and is then treated with an aqueous solution of an alkali hydroxide saturated with a neutral salt of an alkali metal. 10-20 per cent. caustic soda saturated with common salt is the suggested aqueous solution. This solution is separated and washed with petrol to remove any neutral oils. The phenols in the aqueous solution thus obtained are liberated by the known methods such as passing carbon dioxide through it. The yield of phenols is between 10 and 15 per cent. of

the weight of the crude tar and, though considerably less than the phenol fraction obtained by other methods, is very much purer, the phenolic products tending to crystallise without further purification.

A fractionation of the phenolic bodies can be obtained, depending on the fact that a solution of a hydroxide of an alkaline earth saturated with a neutral salt of an alkali or an alkaline earth will extract only the phenols of lower boiling point. The solution obtained after the separation of the pitch is treated with lime water saturated with calcium chloride, the aqueous solution separated and the phenols recovered. The residual solution of phenols is then treated with caustic soda solution saturated with salt as described above and the higher boiling point phenols are recovered from the extract.

The pitch obtained can be emulsified or distilled to yield lubricating and other oils and a harder pitch. W. L.

551. Treatment of Carbonaceous Materials. A. Struben. U.S.P. 1,706,468. Mar. 26, 1929.

The invention relates to the treatment of solid carbonaceous materials and has for its object, the production of products usually obtained by the carbonisation of the material in a retort under dry distillation conditions, but without having resort to such usual carbonisation treatment. The crushed carbonaceous material is mixed with a hydrocarbon carrier having a higher boiling point than that of the products it is desired to obtain. The mixture is introduced into suitable distillation apparatus and subjected to fractional distillation. The carrier may be distilled off from the carbonaceous residue or can be separated by settling or filtration. W. H. T.

See also Abstracts Nos. 285, 298, 313, 323, 332, 357 and 373.

ABSTRACTS.

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Chemistry of Petroleum.

552. CATALYSTS FOR THE FORMATION OF ALCOHOLS FROM CARBON MONOXIDE AND HYDROGEN. IV. Decomposition and Synthesis of Methanol by Catalysts Composed of Zinc and Chromium Oxides. D. S. Cryder and K. Frolich. *Ind. Eng. Chem.*, 21, 9, 867-871.

Further experiments on the decomposition of methanol in the presence of catalysts composed of zinc and chromium oxide indicate that an excess of chromium oxide gives rise to reactions producing appreciable quantities of carbon dioxide and unsaturated hydrocarbons. With catalysts containing an excess of zinc oxide, the main products obtained were carbon monoxide and hydrogen. A sharp minimum in activity of the catalyst occurred when its composition agreed with the formula $C_{75}Cr_{25}$. The relatively constant percentage of formaldehyde produced indicates that it is formed during an intermediate stage in the decomposition of methanol.

Using the same catalysts for the reverse reaction, it was found that the formation of methanol from carbon monoxide and hydrogen agrees very closely with the formation of carbon monoxide and hydrogen in the decomposition experiments. The particular catalyst giving the greatest yield of carbon monoxide and hydrogen in the decomposition experiments was found to give the greatest yield of methanol in the synthetical experiments. Besides yielding further information on the reactions taking place during these processes, and on the behaviour of the catalysts in general, the results obtained agree with previous work using zinc and copper catalysts. Moreover, the results demonstrate the suitability of the decomposition method as a criterion in the selection of catalysts for the high-pressure synthesis of methanol.

H. E. T.

553. THE PRODUCTION OF GASEOUS, LIQUID AND SOLID HYDROCARBONS FROM METHANE. Part II. The Action of Spark Discharge on Methane. H. M. Stanley and A. W. Nash. *Journ. Soc. Chem. Ind.*, 48, 32, p. 238–242.

In this paper the authors give the results obtained by subjecting methane to the action of a spark discharge. They show that the action of the spark discharge gives rise to a variety of reactions, the extent and cause of which

are influenced by such factors as voltage and current, electrode material, spark gap, and above all, the time or duration of sparking. The main products of the reaction are :—

I. *Gaseous.*—Acetylene, with smaller quantities of ethylene and cetylene.

II. *Liquid.*—Light oil of a highly unsaturated nature, exhibiting a pronounced tendency to polymerise to resinous substances. Hydrocarbons containing the group $\equiv CH$ appear to be present in this light oil, together with comparatively small amounts of benzene.

III. Chloroform soluble tar, from which naphthalene and acenaphthylene have been isolated.

IV. Resinous substances.

V. Free carbon.

The primary product of the reaction appears to be acetylene, the higher hydrocarbons produced being probably formed by a secondary reaction on the acetylene-hydrogen mixture present. In support of this view is the fact that, when a mixture containing 15 per cent. of acetylene and 80 per cent. of hydrogen is sparked, liquid and solid products similar to those produced during the sparking of methane are formed, while diacetylene is contained in the reaction gas.

Low gas rates, or longer periods of sparking favour the production of liquid and solid products. Higher current discharges result in a greater percentage conversion of methane into other substances, while at the same time tending to produce greater quantities of light flocculent carbon. The use of electrodes of various materials did not materially affect the course of the reaction, while the same result was obtained by increasing the pressure.

H. E. T.

554. Improvements in the Catalytic Production of Methanol. Commercial Solvents Corporation. E.P. 272864. Oct. 22, 1928. Appl., April 20, 1927.

This invention relates to the production of methanol by the high pressure catalytic combination of oxides of carbon with hydrogen, together with the preparation and employment of improved catalysts in the process. As examples of the catalysts employed in this process may be quoted the following :—

Zinc oxide—chromium oxide—zinc chloride.
Zinc oxide—chromium oxide—vanadium chloride.
Zinc oxide—manganese oxide—magnesium chloride.
Magnesium oxide—vanadium oxide—magnesium fluoride.
Magnesium oxide—chromium oxide—chromium chloride.
Strontium oxide—chromium oxide—zinc chloride.
Zinc oxide—vanadium oxide—magnesium chloride.

Catalysts of this type are most efficient if the oxide of the second group metal is present in predominating quantity over the other promoting oxide.

The process comprises passing a mixture of carbon oxides and hydrogen at a pressure exceeding 50 atmospheres and at an elevated temperature over one or other of the above catalysts.

H. E. T.

555. Manufacture of Cyclic Ketones. I. G. Farbenindustrie A.-G. E.P. 274095. Oct. 8, 1928. Appl., July 7, 1927.

This invention is an improvement of a previous specification, No. 273,321. According to the present process, cyclic ketones are produced by condensing with the aid of an acid condensing agent, hydrocarbons or their derivatives or their substitution products. Intermediate products such as keto-carboxylic acids having an open chain may also be isolated. The cyclic ketones obtained

by this method differ from those of the previous specification by dissolving in sulphuric acid with only a slight fluorescence. They are intended to be used as parent compounds in the manufacture of dyestuffs. Examples illustrating the process are quoted. H. E. T.

556. Manufacture and Production of Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenind. Aktien. E.P. 312717. June 6, 1929. Appl., March 10, 1928.

The invention relates to improvements in the production of hydrocarbons by treating oxides of carbon with hydrogen or gases rich in hydrogen. In order to obtain continuous high yields of hydrocarbons with more than one carbon atom in the molecule, the patent specifies the carrying out of the treatment in the presence of a catalyst. The latter should contain in addition to a metal of group 8 of the periodic system an amount of an alkali compound equivalent to less than about 0·6 part by weight of alkali reckoned as alkali metal per 100 parts by weight of the metal of the eighth group of the periodic system. W. H. T.

557. Catalytic Conversion of Mixtures of Carbon Monoxide and Hydrogen into Compounds Containing More than One Carbon Atom. J. Y. Johnson, Assr. to I. G. Farb. Akt. Ges. E.P. 313,467. Feb. 9, 1928.

The process is an improvement of E.P. 266,405, in which methanol, ethyl and higher alcohols, saturated and unsaturated ketones and other organic bodies are obtained from mixtures of CO and H_2 in the presence of a catalyst. It is found that better results are obtained when the gases are mixed with inert gases such as CH_4, C_2H_6, N_2 or rare gases to the extent of 40 per cent., whereby the development of excessive heat and the deposition of carbon on the catalyst is prevented. The excessive heat may also be partly eliminated by the employment of heat absorbing media, as for example, by disposing the catalyst in tubes bathed by the cold gases before reaction. The lowering of the partial pressure of the CO by the addition of the extraneous gases may be counteracted by raising the total pressure. Elevated pressures from 20 to 1000 atmospheres may be used and, as a rule, the higher the pressure the higher will be the molecular weight of the products obtained. Liquid hydrocarbons are obtained with the aid of catalysts containing iron and cobalt; acids, esters, ketones and the like are obtained with catalysts containing iron, cadmium and copper or potassium, zinc and chromium or zinc chromium and manganese. A catalyst containing iron (100 parts), copper (100 parts), and potassium (0·5 parts as carbonate) will work for a long period at 290° C. and 50 atmos. pressure on a gas mixture containing CH_4 (65 per cent.), N_2 (10 per cent.), CO (10 per cent.) and H_2 (15 per cent.), whereas it rapidly becomes useless owing to deposited carbon when a gas mixture containing CO (40 per cent.) and H_2 (60 per cent.) is used. The products are liquid and gaseous hydrocarbons. Circulating systems are used in which the CO and H_2 are replaced in the gas mixture as quickly as they are removed. T. A. S.

558. Manufacture and Production of Valuable Unsaturated Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenind. Akt. E.P. 315,249. July 10, 1929. Appl., April 10, 1928.

Higher olefines and diolefines, mainly gaseous, are produced from lower members of the olefine series by exposing the initial gases, at any convenient pressure and at a higher rate of flow than that required for the production of acetylene, to the action of high-temperature electrical discharges. Spark discharges or an electric spark may be employed. The electrodes may suitably be of copper, nickel, iron, nickel alloy, etc., and may be cooled

Thus, 80 litres of a gas containing 97 per cent. of ethylene are passed in a continuous cycle and at about 20 litres per hour through 8 cm. spark discharges between copper electrodes, generated by an induction machine having about 60 watts primary consumption. After four cycles, a gas is obtained having the composition: 25·8 per cent. of hydrogen, 0·8 per cent. of methane, 2·8 per cent. of nitrogen and 70·6 per cent. of hydrocarbons $C_n H_m$. The latter have the composition (by weight): 66·2 per cent. ethylene, 13·4 per cent. of propylene, 1·8 per cent. of butylene, 11·4 per cent. of butadiene and 7·2 per cent. of acetylene. About 0·5 gm. of a brown mass resembling cuprene is also formed. A gas containing 97 per cent. of ethylene is passed at 40 litres per hour through an arc generated between carbon electrodes and consuming about 200 watts. The resulting gas contains 29·9 per cent. of hydrogen, 3·6 per cent. of methane, 2·3 per cent. of nitrogen and 64·2 per cent. of hydrocarbons $C_n H_m$. The latter contain (by weight): 69·5 per cent. of ethylene, 15·0 per cent. of propylene, 1·8 per cent. of butylene, 5·7 per cent. of butadiene and 8·0 per cent. of acetylene. W. S. N.

559. Desulphurisation of Hydrocarbons. J. Y. Johnson, Assr. to I. G. Farbenind. A.G. E.P. 315,439. July 15, 1929. Appl., Jan. 14, 1928.

The patent describes a process for the desulphurisation of crude hydrocarbons, which consists in treating them in the liquid or in the vapour phase with hydrogen at elevated temperatures and pressures. The reaction is carried out in the presence of stable metallic sulphides. Hydrogenation of the hydrocarbons is avoided by operating at temperatures ranging between 200° and 300° C. and at pressures lower than those at which hydrogenation occurs. The metallic sulphides can be employed either singly or in combination with one another or with metallic activators and, if desired, deposited on carriers. An amount of hydrogen is employed for the desulphurisation which is in excess of that required for the purpose. The hydrogen sulphide contained in the reaction products is removed, and the gaseous and vaporous products hydrogenated in any known manner. W. H. T.

560. Manufacture and Production of Butadiene. J. Y. Johnson and I. G. Farbenindustrie A.-G. E.P. 315,595. July 18, 1929.

Good yields of very pure butadiene are obtained by treating 1·3-butylene glycol, preferably in the vapour state, with red phosphorus carried upon another solid substance which may be either a catalyst or a carrier. When admixed with a catalyst, *e.g.*, sodium phosphate or potassium aluminium sulphate, the red phosphorus has an activating effect. Such catalysts may also be employed deposited on carriers, *e.g.*, pumice or porcelain balls. Ordinary red phosphorus or the bright red modification may be used. The vaporous glycol may be employed alone or may be mixed with other gases or vapours, *e.g.*, nitrogen, and the process may be carried out at ordinary, increased or reduced pressure. The process in usually operated at 250–350°, particularly good results being attained at about 275–325°.

Thus excellent yields are obtained of reaction products containing 98–99 per cent. of butadiene by treatment of 1·3-butylene glycol vapour (1) at about 300° with granular pumice charged with about 1 per cent. of ordinary red phosphorus, previously washed with caustic soda and dried, (2) at about 250°–300° with monosodium phosphate activated by the addition of about 5 per cent. of red phosphorus, and (3) at about 270° with potassium aluminium sulphate activated with about 5 per cent. of red phosphorus. W. S. N.

561. Improvements in the Manufacture and Production of Diolefines. J. Y. Johnson. E.P. 317,359. Aug. 13, 1929. Appl., Feb. 13, 1928.

The improvement consists in coating the inner surface of the decomposition apparatus employed in the pyrogenic production of diolefines with firmly adhering melts or sinters of inorganic compounds, whereby the undesirable formation of carbon deposits is avoided. The coatings may consist of salts of alkali and alkaline earth metals which melt at temperatures higher than those required in the pyrogeneous process itself. Silicates, phosphates or borates may be employed for this purpose. The coating is produced by spreading the inorganic compound over the inner surface of the metal composing the reaction vessel, drying, and then slowly melting the coating while rotating the vessel in order to obtain a uniform distribution. It is essential that the inner surfaces of the apparatus are kept free from metals of the iron group, since these metals are responsible for the deposition of carbon. By this means the pyrogenic production of diolefines such as butadiene, isoprene or the like, from cyclic or acyclic paraffins by decomposition at temperatures ranging from 500° C. to 600° C. is greatly improved. H. E. T.

562. Treatment of Liquid Hydrocarbons by the Electric Arc. Y. Mercier. E.P. 316,352. July 22, 1929. Appl., April 20, 1928.

The oil to be treated is contained in a vessel provided with two series of electrodes; the stationary electrode of each pair is hollow, and has a conical end extending around the conical point of the adjustable electrode, thereby spreading the arc and avoiding carbon deposition. Oil is fed in through the stationary electrode and the gases formed (chiefly hydrogen and acetylene) are rapidly cooled by the oil and removed, this condition being favourable to acetylene formation. Oil is drawn off from the upper region and recirculated, gases being withdrawn from the top of the vessel under reduced pressure.

The electrodes are separated either by hand or automatically to the maximum extent consistent with stability of the arc. By providing a polyphase arc between a plurality of electrodes the stability may be increased. L. H. A.

563. Manufacture of Unsaturated Hydrocarbons. Society of Chemical Industry in Basle. E.P. 298,090. July 25, 1929. Appl., Oct. 1, 1928.

To avoid the danger from acetylene, which is explosive when under pressure, the reaction between methyl alcohol vapour and an alkaline earth carbide, *e.g.*, calcium carbide, is carried out at ordinary pressure and at 200°–300° C. The mixture of gas and vapour so produced is then treated for separation of the desired products, *e.g.*, by condensation, either directly or after being passed in contact with a heated catalyst. The uncondensed gas, *e.g.*, acetylene, is collected or allowed to escape, wholly or in part, or is returned by a circulating pump to the heated reaction vessel or to the vessel containing the contact mass, for production of unsaturated compounds of high b.p. Contact masses mentioned are carbides, metals, metallic oxides, metallic hydroxides, salts, carbon silicates or other temperature-resisting materials. W. S. N.

564. Polymerisation of Olefines. F. Hofmann, M. Otto and W. Stegemann. E.P. 313,067. June 5, 1929.

The polymerisation of olefines by means of boron fluoride (*cf.*, E.P. 293,487) is accelerated by the use of moist olefines, or by the addition of halogen acids, or of alkyl or aryl halides which split off their halogen in the Friedel-Craft reaction.

Thus moist ethylene is liquefied in an autoclave in the presence of 10 per cent. of boron fluoride and the vessel raised to room temperature. The

pressure rises to about 100 atm. and falls after three to four hours to 15–20 atm. The yield is then 90–95 per cent. of polymeride. The same yield is obtained in three to four hours using 10 per cent. of boron fluoride and 2–3 per cent. of hydrofluoric acid. Ethylene, 10 per cent. of boron fluoride and 12 per cent. of hydriodic acid give a 75 per cent. yield of oil after 10 hours with 100 atm. initial pressure. Ethylene, 10 per cent. of boron fluoride and 5 per cent. of ethyl fluoride give a 100 per cent. yield of oil after 10 hours with 130 atm. initial pressure; a 95 per cent. yield is obtained if ethyl chloride is used in place of ethyl fluoride. Moist propylene, similarly treated, yields 70–75 per cent. of polymeride; in the presence of 3 per cent. of boron fluoride and 1 per cent. of hydrofluoric acid the yield after one hour is quantitative. *Cyclo* Hexane with 6 per cent. of boron fluoride and 1 per cent. of hydrofluoric acid gives a quantitative yield of polymeride. W. S. N.

565. Method of Transforming Olefines into Alkylene Chlorhydrins by Means of Chlorine Water. Th. Goldschmidt A.-G. E.P. 293,754. July 18, 1929. Appl., July 3, 1928.

This invention relates to a method of transforming olefines into alkylene chlorhydrins by means of chlorine water. The apparatus used to carry out this process comprises a collecting vessel supplied with a certain quantity of water. A portion of this water is continuously removed by a pump and then led back to the collecting vessel. Chlorine is introduced into the circulating water at the suction side of the pump, and is so intimately mixed with the water in the suction conduit that it completely dissolves. The liberation of chlorine as gas is prevented since the liquid is under a certain pressure. At the delivery side of the pump the olefine laden gases are forced into the delivery conduit. By means of the rapid flow of the liquid, the gases mix with the liquid forming a froth in the delivery conduit, so that the transformation is affected in the minimum possible time, and is completely terminated when the liquid flows back into the collecting vessel. The residual gases freed from olefines are led off from the collecting vessel for further use. In this manner an extremely simple method of operation is achieved, which is practically automatic and gives satisfactory yields of chlorhydrin.

H. E. T.

566. A Method of Substituting Alkyl Groups in the Aromatic Nucleus. A. Verley. E.P. 288,122. Oct. 16, 1928. Appl., June 16, 1927.

A new process is described whereby alkyl groups may be substituted in the aromatic nucleus. The equation representing the reaction employed is:—

$$C_6H_6 + R{-}O{-}SO_2{-}OH = SO_4H_2 + C_6H_5{-}R.$$

In order to produce this reaction exclusively, the alkyl-sulphuric acid must be dissolved in sulphuric acid monohydrate which is free from pyro-sulphuric acid. In order to produce in actual practice a mixture consisting of $R{-}O{-}SO_2{-}OH$ and sulphuric acid free from the pyro-acid, the following procedure is adopted:—

Twenty-five parts of either potassium or sodium pryo-sulphate are dissolved with heat in 75 parts of commercial 66° Bé. sulphuric acid. The resulting mixture which becomes pasty on cooling, does not contain any free pyro-sulphuric acid and behaves in the same way as sulphuric acid monohydrate as a sulphonating agent. If any alcohol (primary, secondary or tertiary) is added to the sulphuric mixture of pyro-sulphate, alkyl sulphuric acid is formed with elimination of water. This mixture when brought into contact in an agitator with the benzene nucleus in which substitution is to be carried

out, reacts readily at temperatures varying between 30° and 80° C., without any appreciable sulphonation of the nucleus. This reaction is quite general and takes place in practically all cases where actual sulphonation is possible, for example, with hydrocarbons, halogen substituted hydrocarbons, phenolin others, ethers, aromatic acids, etc. The rule governing the position of the substituted alkyl group, is the same as the one governing the substitution of other groups, particularly the sulphonic group. H. E. T.

See also Abstracts Nos. 667 and 668.

Crude Oil.

567. The Continuous Distillation of Tars or Crude Mineral Oils with Superheated Steam. *Chem. et Ind.* 1929. 21, 3, 466–472.

By appropriate treatment of the crude oils obtained by distillation of lignites or shales, a series of products of commercial value may be obtained. The starting point of the treatment of these oils is usually a primary distillation, and on this distillation depends largely the future treatment and value of the products. The object of this paper is not to detail different industrial methods of distillation or to compare them, but to draw attention to the Ab der Halden process in which superheated steam is used to distil the volatile components from the pitchy residues. The theory of distillation with steam is explained and the governing laws are quoted.

In ordinary processes of distillation, the oil is progressively heated to a higher temperature as higher fractions are collected, but in the Ab der Halden process the whole of the volatile fractions are almost immediately vaporized and separated later by fractional condensation.

In coal tar distillation, the residue (pitch) easily decomposes at a temperature of 400° yielding coke which deposits on the walls of the apparatus and hinders the transmission of heat. In this process, the pitch is not heated so highly and is continuously discharged.

The crude tar is pumped through a heat exchanger, heated by hot pitch leaving the retort, and raised in temperature to about 70° C. It is further preheated by passing through a coil of tubes situated in the furnace flue up to 100° C., when the contained water vaporizes and mixture of hot tar and steam enters the retort. This mixture is introduced into the body of pitch contained in the retort, and superheated steam is simultaneously blown into the bottom of the still, which is maintained at a temperature of 290 to 300° C., depending on the nature of the pitch. The pitch overflows continuously through the heat exchanger to storage, whilst the vapours pass overhead into two columns working in series. Each column has a dephlegmator regulated from outside to control the temperature and the composition of the outflowing condensate. The first column separates anthracene oil boiling from 280° to 360° C., whilst the second column separates heavy oils boiling between 230° and 280° C. The vapours containing steam and lighter oils pass to a water condenser, where most of the injected steam and naphthalene condenses. The temperature is controlled so that only the light oil vapours escape. These are rectified in another column and finally condensed in a cooler. The naphthalene and light oils are separated from condensed water in two separators provided.

The fractionating apparatus is thus regulated independently of the distilling section of the plant.

The apparatus for the treatment of low temperature tars is modified for the recovery of phenol. In this case a fractionating tower is used from which different cuts may be drawn off. Dilute caustic soda is introduced

at the top of the tower. The solution of phenates leaving the tower is decomposed with CO_2 liberating the phenols, and the sodium carbonate solution is causticised by lime in a separate part of the plant, then returned to the system.

Finally, the Ab der Halden process may be used for mineral oils. It is fitted with two stills, in one of which a primary distillation to separate the spirit and burning oil is carried out; in the other the operation is completed for the recovery of paraffin and gas oils. Rectifiers and fractional condensers separate the different fractions. R.P.

568. Method of Separating Solids from Oil. J. C. Deacon. U.S.P 1,713,117. May, 14, 1929. Appl., Dec. 10, 1924.

The invention consists in establishing a body of water with a scum at its surface and continuously discharging oil, contaminated with water and solids, upwardly through the water. The layer of scum acts as a filter and separates the water and solid matter from the oil. The solids are continuously added to the scum, and replace those solids that absorb water and sink to the bottom of the water layer.

The water layer is provided with heating coils so that it may be maintained at any desired temperature.

Chemicals, to assist precipitation of the contaminating solids, are added before the oil is admitted to the separating tank. If the solid particles are negatively charged, aluminium potassium sulphate or lime is added and if positively sodium hydrate. W. L.

569. Apparatus for Treating Oil. C. P. Dubbs, assignor to Universal Oil Products Co. U.S.P. 1,690,997. Nov. 6, 1928. Appl., July 9, 1917.

The still consists of a cylindrical upper drum and two lower drums. Each of these lower drums is connected to the upper one by a system of vertical pipes, and the two pipe systems are separated by a fire arch so that the heating gases are compelled to pass up over the arch and then down to the stack.

On the upper end of the upper drum is mounted a combined reflux condenser and heat interchanger. Incoming crude passes through this interchanger, and then to one of four insulated supply drums which may be interconnected in series or parallel.

From here it is pumped into one of the lower drums, and circulates up the pipes to the upper drum, down to the second lower drum and back to the supply drum. Vapours from the still pass through the reflux condenser, and any partially converted vapour falls back into the still and is recycled. Unvaporized oil, which passes through the retort, is, of course, returned to the supply drums and recirculated through the system. W. L.

570. Producing Gasoline and Other Light Hydrocarbons from Heavier Hydrocarbons. B. Ormont. U.S.P. 1,691,300. Nov. 13, 1928. Appl., Oct. 9, 1923.

Oil is heated in a coil disposed in the upper portion of a furnace for the vaporization of the lighter fractions, while water is simultaneously heated in a lower coil for the generation of steam. A vertical connection between the coils allows unvaporized oil to flow by gravity from the upper to the lower coil. Here it encounters a zone of nascent steam, and in this zone vaporizes at a temperature well below its normal boiling point. The resultant mixture of oil vapour and steam passes upward through the vertical connection and thence to a heating coil for further treatment.

It is claimed that by regulating the proportion of oil to water, so that the total latent heat of vaporization of the oil is substantially equal to that of the water light hydrocarbons, such as gasoline, are produced, but if the ratio of oil to water is such that the latent heat of the oil vapour present is less than the latent heat of the water vapour present, then lubricating oils are produced. W. L.

571. Obtaining Light Hydrocarbon Oils from Tar Sands. A. Rule, H. G. Watts and Imperial Chemical Industries, Ltd. E.P. 317,346. Aug. 12, 1929. Appl., May 11, 1928.

Bituminous material, extracted from the tar sand by means of light hydrocarbons, is converted into valuable light oils (b.p. 0°–210° C.) suitable for use as motor fuels and middle oils (b.p. 180°–280° C.), by destructive, high pressure, non-catalytic hydrogenation. Part of the light oil so produced may be used advantageously for subsequent extractions, the solvent being distilled off prior to hydrogenation. Middle oil may be used as a solvent, in which case the whole extraction solution is subjected to hydrogenation, the latter being so controlled as to furnish light oils and middle oils continuously, unconverted residue being returned to the hydrogenation process. The middle oil may be separately cracked or destructively hydrogenated to furnish further light oils. L. H. A.

See also Abstracts Nos. 631, 632, 633, 636, 640, 665, 669, 706, 708, 709, 710 and 711.

Natural Gas.

572. Utilization of Waste Natural Gas. L. G. E. Bignell. *Oil and Gas J.*, 1929, **28**, (9), 39, 148.

At the present time the principal uses of natural gas are as a fuel and as a source of carbon black. The latter use is essentially wasteful, the average yield of carbon being only about 5 per cent. of the theoretical, but the development of the thermal decomposition process to give a good grade carbon black and with high recovery coupled with the production of cheap by-product hydrogen indicates one outlet for an economical use of dry gas. Another important probable use is the production of methyl alcohol, ammonia or automobile fuel by synthesis from water gas derived from steam and natural gas. The pyrolysis of natural gas is a third type of possible reaction for its economic utilization. In addition to the benzol there are unsaturated gases in the residue gas from which such compounds as ethylene glycol could probably be prepared.

If natural gas is combined with air in the presence of a catalyst, formaldehyde, formic acid and methyl alcohol can be produced. A further economic possibility is the conversion of natural gas by chlorination to organic intermediates for use in the manufacture of synthetic resins, refrigerants, dyes, etc. The production of unsaturated liquid products by subjecting natural gas to action of electrical discharge is mentioned as is also the formation of metallic carbides by the interaction of gas and metals at high temperatures with the subsequent production of hydrocarbons by treatment of the carbides with water or acid. W. L.

573. The Thermal Treatment of Natural Gas. Chamberlin and Bloom. *Div. of Ind. and Eng. Chem.*, A.C.S. Meeting, April, 1929.

Aromatic hydrocarbons and olefines have been formed from natural gas by thermal treatment at temperatures below 900°C. Fused silica, clay,

copper, and monel metal are materials which do not catalyse methane and ethane to any great extent. Copper, however, gives the highest yields at the lowest temperatures, but the metal is consumed after a very short time in use. Silica gives high yields at intermediate temperatures and is the most important material, as it is not changed by heat or by-products of the thermal treatment. Iron and nickel readily decompose methane into its elements and are themselves disintegrated by the reaction. Various forms of carbon convert methane into its elements, but that which is the result of the decomposition of natural gas at temperatures from 450° C. upwards has a selective activity in the conversion of paraffins to aromatics, etc. This activity is easily destroyed by partial oxidation or the formation of carbon-metal compounds, graphite being the final result of this loss in activity. By a comparison of these treatments with that of gas manufacture in by-product coke ovens, assuming that the gases have reached equilibrium, the practical yield of benzol from natural gas amounts to 0·2–0·3 gals. per 1,000 cu. ft. of gas treated. The optimum temperature of benzene formation varies for each material and the method of carbon formation thereon. When the concentration of hydrogen has reached 50–60 per cent. of the volume of gas being treated, the oil yield becomes negligible. W. H. T.

574. Oil to Gas Ratios in Natural Gasoline Absorption Plant. E. R. Cox and M. L. Arnold. *Refiner*, 1929, **8**, 8, 63–69.

The object of the investigation was to find a relationship between the oil and gas ratio together with the other working conditions of an absorber, and the efficiency of the absorber. To do this, a formula was worked out which would theoretically express this relationship, secured as much as possible on the active performance of absorbers. The data was checked against the formula, and finally a modified formula was arrived at to express the performance investigated. Raoult's Law is taken as a basis for this formula. If the mol. fraction of a component is taken as X_n and the pressure of that component is P_n, the partial pressure will be X_nP_n in the liquid. In the gas, the partial pressures are divided in accordance with Dalton's Law. If P is the absolute working pressure and Y_n a component of the gas by volume, then the partial pressure is Y_nP. If the liquid is in perfect equilibrium with the gas then $X_nP_n = Y_nP$. (1).

$X_n = L_n/L$ and $Y_n = V_n/V$ where L_n and V_n are the number of mols. of component "n" in the liquid and gas respectively and L and V are the total mols. of liquid and gas. Substituting these values in equation (1)

$$\frac{L_nP_n}{L} = \frac{V_nP}{V}$$

multiplying both sides by L/V_n P_n it becomes

$$\frac{L_n}{V_n} = \frac{LP}{VP_n} \qquad (2)$$

This is a useful equation and applies to any process where the total liquid plus vapour is constant throughout and the two are in equilibrium, *e.g.*, flash distillation and most cases of condensation.

In the case of absorption, P is known and P_n, the vapour pressure of each component can be obtained from working temperature and a chart or table. Then, as L/V is the ratio of total liquid to total vapour, L_n/V_n for each component can be calculated. In a perfect absorber, the wet gas entering and the fat oil leaving the absorber would be in perfect equilibrium and L_n/V_n would be the recovery ratio of each component. For example, if L_n/V_n were

found to be 0·3 for propane, it would indicate a 30 per cent. recovery of propane from the gas. If the value of this fraction is greater than unity it means that the oil circulated cannot be saturated with the component at the given temperature and pressure. To render this a convenient working formula it is necessary to translate L/V into terms of oil gas ratio. This ratio is expressed as gallons (Amer.) of lean absorbent oil per thousand cubic feet of wet gas. Calling this R_o, the weight would be $8{\cdot}33\ R_oG_o$ where G is the Sp. Gr. of the oil and since the mol. is "$_m$" lbs. where "$_m$" is the molecular weight of the substance the mols of the absorbent oil would be $8{\cdot}33\ R_oG_o/_m$. The gasoline absorbed "A," in gallons per thousand cu. feet is $8{\cdot}33\ A\ G_a/_{ma}$ in mols. Hence $L = 8{\cdot}33\ R_oG_o/_{mo} + 8{\cdot}33\ AG_a/_{ma}$.

The mols. of gas are obtained from Avogadro's Law, namely, that the densities of all gases at a given temperature and pressure are proportional to their molecular weights. Hence, at the same temperature and pressure a mol. of gas is a constant volume. At 60° F. and atmospheric pressure, this constant is approx. 379 cu. ft. or $V = 1000/379 = 2{\cdot}4$ mols. Hence L/V

$$= \frac{8{\cdot}33\ R_o\ G_o/_{mo} + 8{\cdot}33\ AG_a/_{ma}}{2.4}$$

$$= 3{\cdot}153\ (R_oG_o/_{mo} + A\ G_a/_{ma})$$

If we call the recovery fractions (F_1 F_2 F_3—F_n)
the formula becomes $F_n = 3{\cdot}153\ (R_oG_o/_{mo} — AG_a/_{ma})\ P/p$.
To calculate the total absorption term $AG_a/_{ma}$ three methods are offered.

(1) Where wet and dry gas tests are available. Since the proportion of methane absorbed is practically negligible it may be assumed that there is the same amount of methane in the dry gas as in the wet gas. Let W be the total wet gas, D the total dry gas, W_1, and D_1, the methane analysis, and the total absorption in cu. ft. A_o, then the latter is the shrinkage or $A_o = W — D$

Equating these and combining we have

$$A_o = \frac{(D_1 - W_1)\ W}{D_1} \quad \text{or for } W = 1{,}000 \text{ cu. ft.}$$

$$A_o = \frac{1{,}000\ (D_1 - W_1)}{D_1} \qquad (4)$$

To express this in gallons per thousand :—

$$A_o = \frac{8{\cdot}33\ AG_a}{ma} \times 379$$

$$\frac{AG_a}{ma} = \frac{{\cdot}316(D_1 - W_1)}{D_1} \qquad (5)$$

(2) Where no analysis is available, assume that the vapour pressure of the total absorption is approximately that of propane then

$$\frac{AG_a}{ma} = \frac{CR_oG_oP}{mo\ ps} \qquad (p3 = \text{vapour pressure of propane}) \qquad (6)$$

Using the data from absorption test tables given and calculating C, the mean value is found to be 0·323 which is probably accurate enough for estimating.

(3) $R_oG_o/_{mo}$ is usually 8 or 10 times as large as $A\ G_a/_{ma}$, hence a small error in estimating the latter does not greatly affect the result.

If we assume a constant ratio between them, the general formula is simplified. The mean value of R_oG_o/m_o for all tests was 0·1716 and the mean value for AG_a/m_a was 0·0216 or $AG_a°m_a = 0·126\ R_oG_o/m_o$ (7) substituting this value in Eq. 3

$$F_n = \frac{3·55\ R_oG_oP.}{m_o\ p_n} \quad (8)$$

This formula is believed to be sufficiently accurate for the usual temperatures and pressures. For extreme cases Eq (6) is preferable.

Forty-five tests were obtained from the principal companies operating absorption plant to test the formula. Tables show the principal data, the analysis of wet and dry gas and the calculated results.

In order to find out what happens at different levels in an absorber tower a series of tests were made on one absorber taking samples of both oil and gas over odd numbered trays from 1 to 15 and the results are charted. As to the type of absorber, no difference is noted between perforated plates and bubble caps. One of each is represented in the two highest tests and both types are also represented in the lowest efficiences. Assuming that the essential validity of the formula (Eq. 3) has been verified by the tests, but that the effect of minor factors such as molecular weight and total absorption may be obscured by other factors, a simplified formula was tried.

For average conditions the simplified formula is quite accurate enough to calculate the oil to gas ratio required to absorb all the normal butane which the particular type of absorber is capable of recovering. The formula is

$$R_o = \frac{75\ p_n}{P}$$

Expressed as a working rule this would be : calculate or estimate the temperature of the oil leaving the absorber. Take the vapour pressure of normal butane at this temperature, multiply by 75 and divide by the working pressure (absolute) in the same units. R. P.

575. Still and Absorption Oil. A. J. L. Hutchinson. *Refiner*, 1929, 8, (5), 84.

The general tendency in absorption plants is to use lighter absorbing oils. These have definite advantages over heavier oils, being excellent absorbing media giving very low pressure drop through heat exchangers and cooling coils and showing high heat transfer values in this equipment. Their disadvantage is the difficulty experienced in recovering the absorbed gasoline without carrying over the light ends of the oil. A saturation test of the lean oil may be very misleading unless it is known what constituents remain in the oil. Examples are given showing that although the enrichment of the oil may be 4·39 per cent. by volume of hexane, the vapour pressure at 80° F. would be about one-fourth that of the oil having 0·962 per cent. by volume of butane alone.

It is also shown that a light absorption oil will give a lean oil of lower vapour pressure to the absorber than will a heavier oil.

It is emphasised that the still is the critical portion of an absorption plant and that given a proper still properly operated, the rest of the plant can be made to operate to its maximum efficiency. W. L.

576. Composition of Natural Gasolines. G. G. Oberfell, R. C. Alden and L. A. Pocock. *Oil and Gas J.*, 1929, 27, (52), 158.

The hydrocarbons present in natural gasoline fall into three groups with relatively distinct boiling point ranges, butanes and lighter; pentanes; and hexanes and heavier. The variations in the proportions in which these groups are present exert a definite and noticeable effect on the course of the Engler distillation curve.

The percentage off at 100° F., corrected for distillation loss was found to be the Engler point which bore the best relationship to the content of butanes and lighter. The percentage of pentanes is given by the relationship of two points on the Engler curve. These points are the percentages off at 100° F. and 140° F. corrected for distillation loss.

These relationships are presented graphically. The greatest source of error resides in the fact that Engler distillations cannot be performed without some loss. If the sample is cooled before pouring and the distillation carried out with all care at a normal room temperature it is claimed that the method gives reasonably accurate results in all instances. W. L.

577. The Sampling of Gas over Mercury at Constant Rate. S. Pexton and W. K. Hutcheson. *Jour. Soc. Chem. Ind.*, **48**, (**32**), 242–244.

An account is given of an ingenious arrangement, designed with the object of collecting a sample of gas over mercury, continuously and at a constant rate. A mathematical theory of the instrument is developed. H. E. T.

578. Fractional Analyses of Natural Gas. W. J. Podbielniak. *Oil and Gas J.*, 1929, 27, (52), 30–113.

An important application of the fractional distillation method of analysis is the analysis of dry gases from a gasoline plant to determine the amount of valuable gasoline hydrocarbons passing away from the plant. Normally, this is of the order of 0·1 to 0.2 per cent. and consequently the demand on the analytical method is unusually severe.

The apparatus described in Abstract No. 342, 1929, is used. The gas sample is cooled just before its admission to the distilling bulb by passing through a gasoline bath maintained at a temperature just above the boiling point of methane. A jet of liquid air blowing through a cooling vessel in the bath is the method used for attaining the desired temperature. The rate of admission can be increased more and more so long as the top temperature in the column stays below the boiling point of methane, showing that no ethane and heavier is being lost with the vent gases. In practice it has been found possible to enter 40,000 ccs. of gas sample in half an hour with adequate cooling and without loss of ethane in the vent gas. When sufficient of the sample has been entered, the inlet pipe in the cooling vessel is sealed with mercury, the apparatus disconnected from the sample container and the fractional analysis carried out as usual.

This method minimises the time necessary for the introduction of even a very large sample of dry gas and has been found to be entirely satisfactory for the purposes to which such analyses are put. W. L.

579. Gasoline Plants at Oklahoma City. C. O. Willson. *Oil and Gas J.*, 1929, 28, (9), 37, 148.

This article gives a brief survey of two gasoline plants, working at the Oklahoma City Pool, and dealing with very lean gas. In the Colin plant the gas is first cleared of all entrained matter in a tangential scrubber and then enters the bottom of a 6 by 38 feet oil absorber. Any entrained absorption

oil is removed from the residue gas by another tangential scrubber and the gas is then available as fuel. A liquid level control determines the flow of rich oil in the absorber. The oil passes first to a weathering tank where occluded gases are removed through a back pressure control. From here it is pumped through a pressure heat exchanger, a gravity heat exchanger and finally to a steam preheater. The oil is then discharged in the bottom of a combination evaporator and rectifying column.

Denuded oil in the bottom of the evaporator operates a liquid level control which in turn regulates the pump taking suction from the evaporator. This oil flows through the gravity heat exchanger and here the pump takes suction and discharges through the pressure heat exchangers, oil coolers, finally returning the oil to the top of the absorber. The combination of a liquid level control at the weathering tank and the same control for the denuded oil at the evaporator makes the absorption and distillation operation automatic. In the rectifying column the reflux pump is controlled automatically by the temperature at the top of the column. Vapour from the column passes through a Ventura mist extractor before going to the condensers.

The Foster Petroleum Plant is operated as a combination gas lift and gasoline plant. A back pressure of seventy-five pounds is maintained at the well, giving a plant pressure of about fifty pounds. After passing through scrubbers the gas passes direct to the absorber. The absorption and distillation operation is conventional. The residue gas from the steam still is used in the gas lift operation and is returned to the well at a pressure of 300 lbs. W. L.

580. Water Vapour in Natural Gas. *Oil and Gas J.*, 1929, (28), 9, 38.

The problem of water vapour in natural gas is dependent upon the understanding of several factors: saturation, pressure, temperature, dewpoint, carrying capacity and relative humidity.

A chart is given showing the maximum percentage of water vapour that can be carried by a gas at temperatures ranging from 0 to 140° F. and at pressures from 0 to 600 lbs. per square inch.

It is concluded that the danger of operation troubles is minimised by limiting the amount of water vapour in the gas as it enters the transmission system. In some cases it is possible to fix temperature and pressure conditions in later operation so that the gas is capable of taking up more water rather than condensing the water vapour to water. W. L.

581. Apparatus for Recovering Gasoline. H. B. Bernard, ass. to Sinclair Oil and Gas Co. U.S.P., 1,713,323. May 14, 1929. Appl., Nov. 14, 1924.

The apparatus described comprises an absorber for treating natural gas or gas mixtures with a liquid absorbent, a preliminary still and the necessary connections for charging it from the absorber, an exhausting tower through which the liquid residue from the still is passed in counter current flow and in direct contact with the vapours and gases from the still. Connections are made between the still and this tower including means of regulating and reducing the pressure between the still and the tower. A cooler is provided together with the necessary connections for returning stripped absorbent from the exhausting tower to the absorber. The upper portion of the exhausting tower may be used as a rectifying column or a separate dephlegmator can be installed. It is claimed that the apparatus enables a more complete stripping of the liquid absorbent and the maintenance of conditions in which lower vaporization temperatures can be employed. W. L.

582. HOT PURIFICATION OF GASES. Compagnie Internationale pour la Fabrication des Essences et Petroles. E.P. 298,236, July 25, 1929. Appl., Oct. 5, 1928. Conv. (France), Oct. 6, 1927.

The invention claims a speeding up of the regeneration by air or oxygen of metallic oxides which have been converted to sulphides during the hot desulphurization of commercial gases. Excessive rise of temperature, the consequent fusing of the purifying agent or carrier, and the destruction of the fine state of sub-division necessary for efficient action are avoided by the use of a purifying element in which the ratio of surface to volume is greatly increased. Each purifying element consists of two vertical coaxial tubes leaving between them an annular space closed at each end by annular covers. The purifying mass is disposed in the annular space between two grids. the empty end chambers each having a tangential pipe fitted, provided with a branch pipe and stop-cocks for the regeneration. During regeneration air passes upward, the heat being radiated both toward the exterior and toward the interior of the apparatus.

The concentric cylinders have diameters for example of 300 and 500 mms. respectively, but they may be of any cross-sectional shape desired, while the annular space may be made of minimum width at the zone of maximum temperature, the width increasing progressively away from this zone.

L. H. A.

583. PREPARING HYDROCARBON PRODUCTS. F. A. Howard. U.S.P. 1,727,303. Sept. 3, 1929. Appl., April 25, 1924.

A process is provided for preparing hydrocarbons of low boiling point. In accordance with this invention natural gas or refinery tail-gas, is brought into contact with an absorber menstruum in an absorber, a compressor being employed if higher compression of the gas is desired. The denuded gas leaves the absorber through a pressure control valve into the gas line. The absorbing material which is preferably a mineral seal petroleum oil, takes up available hydrocarbons from the gas, and is then passed to a stripping still where it is heated in order to recover the absorbed constituents.

H. E. T.

584. IMPROVED PROCESS FOR THE PRODUCTION OF HYDROGEN AND GAS MIXTURES CONTAINING THE SAME. J. Y. Johnson. E.P. 307,529. Mar. 4, 1929. Appl., Dec. 2, 1927.

This invention provides a process for converting solid, liquid or gaseous carbonaceous substances containing hydrogen, into a gas consisting of a mixture of hydrogen and nitrogen suitable for the synthesis of ammonia. The gases are exposed to the action of an electric arc, while they are in association with air or oxygen. The resulting carbon monoxide is converted into carbon dioxide by means of steam, and then the resulting hydrogen-nitrogen gas mixture is freed from the carbon dioxide thus formed. As an example of this process the following is given :—

Natural gas containing about 96 per cent. of methane, is mixed with air in the ratio of either 1 to 5 or 1 to 7, and this gaseous mixture is subjected to the action of the electric arc. A gas is thus obtained containing about 77 per cent. of nitrogen, 10 per cent. of hydrogen, 9 per cent of carbon monoxide and 4 per cent. of carbon dioxide. After treatment with water vapour in order to convert the carbon monoxide into carbon dioxide, and then removing the carbon dioxide the resulting gas contains about 80 per cent. of nitrogen and 20 per cent. of hydrogen.

H. E. T.

585. Improvements in the Desulphurisation of Gases. J. Y. Johnson, Asst. to I. G. Farbendind, A.G. E.P. 312769. Jnne 6, 1939 Appl., April 26, 1928.

The patent describes a process for freeing industrial gases such as water gas, producer gas, etc., from dust and sulphur compounds. Air or other oxygen-bearing gases or oxygen, and ammonia are added to the gas while still hot and laden with dust, the resulting sulphur and dust being deposited from the current of gas, a temperature of between 100° to 300°C. being maintained. W. H. T.

See also Abstracts Nos. 553, 586, 590, 591, 669, 707 and 723.

Motor Spirit.

586. Volatility Data on Natural Gasoline and Blended Fuels. O. C. Bridgeman. *S.A.E. Journal*, 1929, **25**, 137.

The examination of gasoline volatility in the Sligh equilibrium air apparatus has been extended to temperatures below 0° C. in order to include natural gas gasoline and blended fuels. The method consists in supplying liquid gasoline and dry air in such proportions to a thermostatically controlled vaporizer that a given resultant mixture of gasoline vapour and air is obtained. The volatility is then expressed as the per cent. liquid fuel vaporized at the temperature of the vaporizer.

Since the degree of vaporization is a function not only of temperature conditions but of the rate of flow, the method adopted was to make observations, under given conditions, at varied rates of flow and to extrapolate to zero flow.

Thirteen fuels, including several ethyl ether-gasoline and benzene-gasoline blends were examined, and the ratings at different temperatures compared with the A.S.T.M. distillation curves for the same fuels. The results are shown graphically for a resultant mixture of 16 : 1 air-fuel ratio, but comparable results were obtained for 8 : 1, 12 : 1 and 20 : 1 air-fuel ratios. In all cases the equilibrium-air curves were similar in form to, and rated the fuels in the same order as, the A.S.T.M. curves. For the ethyl-ether blends, the temperatures for easy starting on these fuels are deduced on the assumption that 7·5 per cent. of the fuel is evaporated from a 1 : 1 (air-liquid fuel) mixture, thereby enabling a start to be made in 10 revolutions of the crankshaft. The temperatures so calculated agree fairly well with those observed by Robb (*Aeronautical Journal*, Sept., 1922, 374), on the starting of aero-engines with similar mixtures.

The dew points of the various fuel vapour-air mixtures are deduced by extrapolation to 100 per cent. vaporization. As in the previous work here is found to be a constant relation between the dew point and the A.S.T.M. 90 per cent. point for each fuel. G. B. M.

587. Data from Air Distillation Explained. G. G. Brown. *Oil and Gas J.* 1929. 28, 5, 46, 107.

The volatility of a gasoline is not represented by its endpoint, but total volatility or dew point is closely related to the 90 per cent. point. Generally, partial volatility is more important than total. Equilibrium volatility is the per cent. by weight vaporized under equilibrium conditions at specified temperature, pressure and air-fuel ratio. A brief review of previous work is given. Cragoe and Eisinger suggested the equation.

Dew point ° F. = ¼ (A.S.T.M. 90 per cent. in ° F. — 186) as giving the dew point of a 12 to 1 air-fuel mixture within the limits of plus or minus 6° F. A brief description of the apparatus employed by the writer is given, together with the methods of test and the results obtained, most of which are presented graphically.

Equilibrium volatilities computed from corrected equilibrium vaporization curves agree with experimental air distillation curves at the higher temperatures. Dew points determined by calculation are also in agreement with those obtained experimentally.

Attempts are made to relate a particular point in the A.S.T.M. distillation curve with a similar point on the equilibrium air distillation curve that will fit all fuels, but it is found impossible. If fuels of similar distillation range and characteristics are to be compared, such a relationship may be applied with a reasonable degree of accuracy. Graphs are given which allow the true equilibrium volatility of natural gasoline blends with U.S. motor fuels to be closely estimated from their A.S.T.M. distillation curves. W. L.

588. Treatment of Pressure Distillate. W. R. Hounsell. *Oil and Gas J.*, 1929, **27**, 43, 31 and 147–8.

This article describes the refining of a very sulphurous Dubbs pressure distillate from heavy Smackover crude with "blue solution," which is prepared by precipitating cupric hydroxide with caustic soda in cypress wood tanks, and just redissolving the precipitate in ammonia.

During the cracking process, large amounts of sulphuretted hydrogen and hydrochloric acid gas were produced and to avoid corrosion of equipment, sufficient dilute ammonia solution was introduced into the vapour lines with the pressure distillate to render the latter neutral. The condensate was given a water wash before storing. As the crude distillate contained large percentages of cyclic and unsaturated compounds and much combined sulphur, refining by means of such reagents as acid, hypochlorite and doctor solution alone was found to be unsatisfactory, but treatment with cupric ammonium hydroxide gave a sweet product of high anti-knock value.

The crude distillate was first treated with sulphuric acid (7 lb. to the barrel), water washed, and then thoroughly mixed with blue solution in orifice columns, the amount of the solution being controlled so that the spent liquor, which carried a black precipitate, was still faintly blue, showing a slight excess of the reagent. The treated distillate was redistilled and stored.

Treatment with a large quantity of sulphuric acid of high concentration was avoided, as polymerisation of the olefins present was not desired, but merely the removal of the highly unsaturated hydrocarbons; the undesirable mercaptans were removed by the blue solution, while the naphthenes were unaffected by the mild alkali which was employed. D. G. P.

589. Revised U.S. Motor Gasoline Specifications. W. T. Liegenhain. *Oil and Gas J.*, 1929, **28**, 5, 141–149.

A revision of the U.S. motor gasoline specifications and the establishment of a new grade to be known as motor fuel V have been promulgated by the Bureau of Standards, and the new specifications will be placed in effect on September 11, 1929.

Master specification for motor gasoline; U.S. Government.

This specification covers the grade of gasoline used by the U.S. Government as a fuel for automobile, truck, tractor, motor boat and similar engines.

Corrosion test method 530.22. A copper strip to show not more than extremely slight discolouration when submerged in the gasoline for three hours at 122° F.

Distillation range. Method 100.13.

With 10 per cent. off, the temperature shall not be less than 50° C. nor more than 80° C., provided that for each per cent. distillation loss less than 4 per cent. obtained in the A.S.T.M. distillation, the minimum 10 per cent. temperature requirements shall be lowered by 3° C.

With 50 per cent. off the temperature shall not exceed 140° C., and with 90 per cent. 200° C.

The end point shall not be higher than 225°C., and a minimum of 95 per cent. shall be recovered as distillate.

Sulphur method 520.11. Sulphur must not exceed 0·1 per cent.

Master specification for motor fuel V. This covers a special grade of fuel which may be used by the Government for emergency vehicles, naval and military equipment, etc.

Corrosion test. Method 530.22. Extremely slight discolouration on copper strip when submerged in the gasoline for 3 hours at 122° F.

Distillation range. Method 100.13. With 10 per cent. off, the temperature shall be not less than 50° C. and not more than 65° C., provided that for each per cent. distillation loss less than 4 per cent. obtained in the A.S.T.M. distillation, the minimum 10 per cent. temperature requirement shall be lowered by 3° C. With 50 and 90 per cent. off the temperature shall not exceed 125° C. and 180° C. respectively. The end point shall not be higher than 205° C., and a minimum of 95 per cent. shall be recovered as distillate.

Sulphur method 520.11. Sulphur must not exceed 0·1 per cent. W. L.

590. Production of Liquid Polymerisation Products from Gases Containing Liquid Hydrocarbons. C. Epner. E.P. 294100. June 27, 1929. Appl., June 8, 1928. Conv. (Ger.), July 16, 1927.

This patent relates to the production of liquid power fuels from gases containing hydrocarbons. The gases are led through a Siemen's ozoniser tube and are there subjected to the silent discharge of 8000 volts and 10,000 periods. The rate at which the gas passes through the tube is adjusted in relation to the voltage and frequency employed, having regard to the degree of polymerisation which is desired. For the production of liquid power fuels rapid removal of the products from the electric field is necessary. The reaction is therefore carried out at such a temperature that the products are evolved in vapour form. These are removed from the gas current by condensation, activated charcoal or oil washing.

Part of the products consists of unsaturated products. These may be converted into saturated compounds by hydrogenation, and preferably the hydrogen contained in the residual gas is employed for this purpose, the hydrogenating catalyst being arranged immediately following the field zone. W. L.

591. Improvements in the Manufacture and Production of Aromatic Hydrocarbons. I. G. Farbenindustrie. E.P. 281,298. Nov. 15, 1928. Appl., Nov. 23, 1927.

A process is described for the production of aromatic hydrocarbons such as benzene, naphthalene, anthracene and phenanthrene by treating non-aromatic hydrocarbons under elevated pressures and temperatures exceeding 500° C. in the presence of catalysts, with an excess of hydrogen or gases containing hydrogen in a circulatory system. Care is taken that content of the circulating gases in gaseous hydrocarbons formed during the reaction is kept low, by removing periodically the gaseous hydrocarbons such as methane which are produced. All the materials comprising the apparatus and the catalysts employed are so chosen that they do not produce either coke or

methane during the reaction. Methane, if produced, is removed from the circulating system by means of suitable solvents and absorbers, and may be utilised in the production of hydrogen. Catalysts suitable for this process are chromium in the metallic state or oxide, or chromium, zinc and aluminium alloys or other metallic oxides, and manganese chromate. On cooling the gases leaving the reacting vessel a liquid rich in aromatic hydrocarbons is obtained which may be used as a motor fuel or for solvent purposes.
H. E. T.

592. Obtaining Light Hydrocarbons from Solid or Liquid Fuels. P. Girard, F. Petit and A. Charbonneau. E.P. 299,861. June 27, 1929. Appl., May 16, 1928. Conv. (Fr.), Nov. 3, 1927.

The solid or liquid fuel is distilled at a temperature between 300° C. and 650° C. in the presence of an oxidizing agent, other than a halogen, adapted to yield oxygen to the charge without liberating free oxygen. Potassium permanganate, manganese dioxide and potassium dichromate are mentioned as suitable oxidizing agents.

The reaction is reinforced by ionisation, the latter being effected by means of a high frequency discharge. This discharge takes place between two electrodes located in the path of the gas between the retort and the condenser.
W. L.

593. Purifying Hydrocarbon Oils. L. Link and M. B. Amis, Assrs. to Standard Oil Development Co. June 25, 1929. Appl., Dec. 9, 1925. U.S.P. 1,718,714.

The process of this invention comprises the treatment of corrosive straight run spirit with caustic alkali that has taken up certain constituents of light cracked hydrocarbon distillates. W. H. T.

594. Process of Sweetening Hydrocarbon Distillates. T. B. Kimball. E.P., 291,379. Aug. 8, 1929. Appl., May 25, 1928.

Sulphur compounds (in particular mercaptans) are removed or partly removed from normally liquid hydrocarbons by vaporizing the latter and bringing the vapour into contact with a non-oxidizing alkaline solution, in counter-flow, heated to a temperature above the condensation temperature of the vapour but below the boiling point of the alkaline solution at the pressure employed. The non-oxidizing alkaline solution is normally aqueous sodium hydroxide. W. S. N.

595. Process of Treating Cracked Hydrocarbons. G. Egloff and J. C. Morrell. U.S.P. 1,725,068. Aug. 20, 1929. Appl., July 3, 1926.

In treating cracked pressure distillate or gasoline, one of the usual methods is to treat with sulphuric acid, caustic soda or plumbite. After the chemical treatment the oil is usually redistilled with steam to a finished marketable gasoline.

A more effective removal of the sulphur compounds may be obtained by substituting for sulphuric acid a solution of copper sulphate in sulphuric acid. With some cracked distillates, for example Californian cracked oils, approximately 0·3 lb. of copper sulphate per barrel will desulphurize them so that they pass the commercial tests of doctor and corrosion. R. P.

596. Preparing Hydrocarbon Products. J. Simpson, Assr. to Standard Oil Development Co. U.S.P. 1,718,713. June 25, 1929. Appl., Jan. 30, 1924.

The invention relates to the preparation of petroleum distillates, and more particularly to the refining of light products. The process consists of

subjecting a sour naphtha distillate to the action of a sodium plumbite solution, separating the latter and subjecting a naphtha distillate containing corrosive sulphur to the action of the same solution. W. H. T.

See also Abstracts Nos. 553, 573, 576, 581, 583, 597, 604, 625, 629, 630, 660, 661, 662 and 719.

Kerosine.

597. Method of Treating Petroleum Distillates with Respect to Discoloration. C. H. Jobson. E.P. 312,774. June 6, 1929. Appl., April 30, 1928.

This invention relates to a method of treating petroleum distillates having A.P.I. gravity higher than 37°, with respect to discoloration. It is well known that refined petroleum products, especially gasoline and kerosine, on being stored are apt to develop colour, due to oxidation, which renders the products objectionable to the trade. The process consists of adding to the distillate a minute proportion of an aromatic or aliphatic dihydroxy compound, an aromatic amine, a phenol, urea, urea derivative or an alkaloid, whereby colour formation is prevented or lessened. The proportion of agent should not be greater than 1 part in 10,000 and insufficient in amount to colour the distillate. W. H. T.

See also Abstracts Nos. 604 and 627.

Fuel Oil.

598. Filter for Fine Filtering of Fluids, especially Fuel Oils. T. V. Hemmingsen. E.P. 307,003. April 26, 1928. Conv. (Denmark) March 1, 1928.

A filter for fuel oils for internal-combustion engines consists of a casing having a conical chamber with inlet and outlet connections and a conical filter body located centrally in the chamber so that a narrow passage is produced between the filter body and the chamber. Longitudinal grooves are provided on the conical surface of the filter in two series, one connected to the inlet and the other to the outlet. The oil to be filtered travels through the first series of grooves to the narrow passage between the filter body and the chamber, where filtration takes place, and thence *vid* the second series of grooves to the outlet. G. 8.

See also Abstracts Nos. 643, 644 and 645.

Lubricating Oil and Wax.

599. Some Properties of Fullers Earth. Davis and Messer. A.I.M.M.E. Tech. Pub. No. 207.

Seventeen earths, fourteen of which required acid treatment in order to develop their bleaching power for oils, are the basis of this work. The earths from which good acid-treated oil-bleaching adsorbents may be prepared are, like fullers earth, variable in composition, but their silica-alumina ratio is not so high. The quantity of treating earth required and the conditions necessary to yield the best products have to be determined for each earth. The authors show that whereas one earth may require a certain treatment to make it the

most efficient adsorbent for one oil, another treatment is better when used on another oil. Although most fullers earths may be heated at 650°–700° C. without their efficiency being destroyed, an earth of high magnesia content may lose much of its bleaching power at 450° and still more at 600° C., while an acid treated earth may be come inefficient at 600°. At 900° the bleaching power of all the earths tested was destroyed. Swelling in water seems to be peculiar to earths that become good adsorbents by acid treatment. W. H. T.

600. A New Sulphuric Acid Test for Insulating and Lubricating Oils. Walther. Erdöl u. Teer, 1929.

The use of 96 per cent. sulphuric acid for testing refined oils is of very doubtful value. According to previous experience, it indicates the presence of both the unsaturated compounds which act as protective compounds and reduce the tendency of the oil to oxidise, and the other labile compounds which greatly accelerate the speed of oxidation. For used oils a knowledge of the solubility in 96 per cent. sulphuric acid is valueless unless the solubility of the fresh oil is also known, and in any case the determination has an accuracy of only 0·5 per cent. The action of sulphuric acid on the different groups of compounds contained in the oil varies according to the concentration of acid, and it is upon this fact that the analytical methods of Reisenfeld and Bandte, Kattwinkel, etc., are based. As in the present case only the most labile compounds are concerned, a dilute acid should be used. The oil is diluted with benzine in order to permit the acid layer to settle out quickly. 5 ccs. of oil is measured into a test tube of about 15 mm. internal diameter, dissolved in 5 ccs. of benzine, and 5 ccs. of 50 per cent. sulphuric acid is added. The tube is well shaken for half a minute and then allowed to stand. When separation is complete, the colour of the acid layer, the appearance and amount of the intermediate layer between the acid and oil and in some cases the change in colour of the oil layer are observed. From a consideration of the results from a number of tests carried out as above, it seemed probable that when the acid is added to a used oil, the oxidation products present are separated into two groups ; the acids are dissolved in the sulphuric acid layer, colouring it darker, and the asphaltic substances, if any, are thrown out of the oil to form an intermediate layer between oil and acid and allow some change of colour in the original oil. For the same kind of oil these changes are proportional to the degree of ageing, and therefore it should be possible from accumulated data from such tests to set up standards as limits up to which an oil could be used again or used for a longer period. For example, a transformer oil would no longer be usable when the acid layer became nut-brown in colour or was brown and turpid, or an intermediate layer more than 1 mm. thick was formed. Oils similar to transformer and turbine oils, which must be refined to a high degree, show no changes in the acid layer and no separations. The test can be usefully applied in the process of regenerating or of refining oils. Occasionally unused lubricating oils which had apparently been insufficiently refined showed separation and colouring of the acid layer. The author proposes to test such oils by ageing tests and by practical use and to report his findings. The test proposed is simple and easy to apply, and is calculated to furnish a basis for qualitatively judging an oil as to its degree of ageing and refining. W. H. T.

601. Continuous Rectification of Liquids at High Boiling Point. E. A. Barbet. U.S.P. 1,722,322. July 30, 1929. Appl., June 11, 1923.

The invention relates to the continuous rectification of hydrocarbons and other liquids containing constituents of high boiling point. The material to be rectified is subjected to the indirect action of saturated steam whereby a

portion of its constituents is vaporised. The residual unvaporised liquid is subjected to the indirect action of superheated steam, and by this means substantial vaporisation is effected. The vapours arising from both these treatments are brought into intimate contact in a single rectifying column with a reflux of condensates formed from the vapours. The condensed material is withdrawn as a plurality of sharply defined fractions boiling within different temperature ranges. W. H. T.

602. Production of Lubricating Oil from Gases containing Hydrocarbons. C. Epner. E.P. 294,099. June 20, 1929. Appl., June 8, 1928. Conv. (Ger.) July 16, 1927.

Cracking gases are fed slowly through several Siemens tubes connected in series and subjected to a high-frequency and high-tension discharge. The frequency may be varied from 500 to 10,000 cycles. Tensions over 1,000 volts can be used, but tensions between 6,000 and 25,000 volts are preferable. The type of reaction products obtained depends on the duration of the electrical action, their viscosity increasing with the time. The oils produced are in part unsaturated and may subsequently be subjected to hydrogenation to produce saturated products. W. L.

603. Recovery of Organic Acids from the Oxidation Products of Paraffin Hydrocarbons, Waxes and the Like. J. Y. Johnson, assr. to I. C. Farbenind Akt. E.P. 315,813. July 16, 1929. Appl., Mar. 16, 1928.

Organic acids are recovered from the acid oxidation products of paraffin hydrocarbons, waxes and the like by saponifying with alkaline earths (including magnesia) in the presence or in the absence of water, drying the soap obtained if necessary, extracting the unsaponifiable matter from the comminuted soap with an organic solvent, and decomposing the purified soap with a mineral acid. Thus, an acid oxidation product of paraffin wax containing 36 per cent. of unsaponifiable material is vigorously stirred for two hours at 160° C. in an autoclave at 5 atm. with an aqueous suspension of slaked lime in 8 per cent. excess of the theoretical. After withdrawing the dark aqueous layer the calcium salt is thoroughly dried on a drying roller and extracted at 40° C. with benzine of b.p. 60°–100° C. The purified calcium salt is dried and decomposed with mineral acid giving a quantitative yield of acid constituent containing only 1–2 per cent. of unsaponifiable matter. A crude acid oxidation product containing 29 per cent. of unsaponifiable matter is stirred with twice the theoretical quantity of dry calcium hydroxide and the mixture kept at 135° C. for six hours. The hard brown lime soap is ground to a fine powder and extracted repeatedly with benzine, b.p. 60°–100°C. The purified soap is dried by warming, and is decomposed with mineral acid, giving a quantitative yield of fatty acid containing 2·4 per cent. of unsaponifiable matter W. S. N.

604. Treatment of Emulsion Oils. E. H. Payne and S. A. Montgomery, assrs. to Standard Oil Co. U.S.P. 1,726,309. Aug. 27, 1929. Appl., Mar. 1, 1922.

Emulsion oils are treated with the spent caustic soda or "doctor" solutions resulting from the treatment of oil distillates. The caustic soda alkalinity must have been reduced by at least one-fifth, and preferably should not exceed one-third of the original alkalinity, which may have been 18 to 20 per cent. The emulsion to be broken down is thoroughly mixed with 0·25 to 5 per cent. by volume of spent caustic soda solution at about 180° F., thereafter being allowed to settle at this temperature, when clean-cut separation is claimed to take place. L. H. A.

605. Treatment of Emulsion Oils. E. H. Payne and S. A. Montgomery, assrs. to Standard Oil Co. U.S.P. 1,726,310. Aug. 27, 1929. Appl., Mar. 1, 1922.

Emulsion oils are treated with the spent caustic soda or soda ash solutions resulting from the neutralisation of acid-treated oils and waxes. The alkalinity of the soda ash must have been reduced by more than one-third, and preferably by more than one-half. The emulsion to be broken down is thoroughly mixed with 0·25 to 5 per cent. by volume of settled spent alkali solution at about 180° F., thereafter being allowed to settle at this temperature, when clean-cut separation is claimed to take place. L. H. A.

606. Separating Congealable Solids from Oils. J. F. P. Schönfeld, assr. to N. V. de Bataafsche Petroleum Maatschappij. U.S.P. 1,724,732. Aug. 13, 1929. Appl., Mar. 12, 1925.

The invention claims to separate congealable solids from mineral, animal, or vegetable oils (particularly paraffin wax from a mineral paraffin-containing oil fraction) by atomising the oil into a cool atmosphere containing a fog of water particles. Oil at a temperature 3–10° C. above its solidifying point is delivered through atomisers into a chamber of cooler atmosphere, where it is brought into contact with a cold water fog supplied from a parallel set of atomisers. A quick transmission of heat is obtained from the fine oil particles *via* the air to the water fog. Solidified particles of paraffin are retained on an inclined sieve, and move slowly down to an outlet, thereafter being melted down for further treatment, while the oil percolates through the sieve, being led off separately. This oil may be atomised over again, and a lower melting point paraffin obtained from it. The nature of the solid product is governed by the oil and water temperatures, and by the rate of oil feed. A marked saving of time over the old cooling and pressing method is claimed. L. H. A.

607. Manufacturing Paraffine. C. A. Ward. Assr. to Standard Oil Development Co. U.S.P. 1,718,672. June 25, 1929. Appl., Nov. 20, 1925.

A process is described whereby a wax-containing petroleum fraction, from which paraffin is not normally directly obtained without other preliminary treatment, may be satisfactorily chilled and pressed, thereby yielding a residual oil of high lubricating value. After treatment, the slack wax produced can be readily finished into a paraffin of commercial qualities. The wax-containing fraction is chilled, mixed with a filter-aid such as clay, and pressed in order to remove the slack wax. The latter, being very difficult to press, is subjected to a cracking distillation in order to produce a distillate of sweatable slack wax. W. H. T.

608. Separating Wax from Mineral Oils. R. E. Wilson. Assr. to Standard Oil Co. U.S.P. 1,719,350. July 2, 1929. Appl., July 18, 1922.

The invention relates to a method of removing the amorphous and other paraffin wax from heavy petroleum residues or cylinder stocks for the production of lubricating oils. The process consists of mixing the heavy residue or cylinder stock with an oil-miscible liquid which precipitates the bulk of the wax at a low temperature. The liquid consists essentially of an alcohol of the aliphatic series such as isopropyl alcohol containing between three and seven carbon atoms. The heavy oil or cylinder stock may be diluted with naphtha before being mixed with the precipitating liquid. W. H. T.

See also Abstracts Nos. 612, 625, 626 and 627.

Special Products.

609. Triethanolamine Oleate for Oil Sprays. G. L. Hockenyos. *Ind. and Eng. Chem.*, **21**, vii., 647.

Commercial triethanolamine consists of 70–75 per cent. of triethanolamine, 20–25 per cent. diethanolamine and 0–5 per cent. monoethanolamine, boils at approximately 277° C. at 150 mms. pressure, and is a strongly basic oil, colourless, with an ammoniacal smell and soluble in water. Mixed with oleic acid it forms a stiff jelly which is easily liquefied by gentle heating.

As water is formed by combination of the base with the acid, this is settled out on addition to mineral oil in which the soap is soluble. A water emulsion of the oil-soap solution can be made by direct agitation of the two constituents or by melting the soap and boiling out the water, adding oil and alcohol and clarifying with oleic acid.

Tests have shown that emulsions giving best adhesion on waxy leaf surfaces are obtained if not more than 10–20 parts of mineral oil per one part of triethanolamine and 1.2 pts. of oleic acid are used, and that addition of up to 10 per cent. by volume of carbon tetrachloride or 10 per cent. by weight of *p* dichlorbenzene can be made without etriment to the emulsion as a leaf spray.

The last solution has been found highly effective for killing mealy bug and red spider. A. R. S.

610. Uses of Asphalt Emulsion in Chemical Plant. L. Kirschbraun. *Chem. and Met. Eng.*, 1929, **8**, 477.

It is the purpose of this article to discuss briefly the properties and uses of stabilized asphalt emulsions. Asphalt emulsions require no heating, and it is necessary only to permit the coated material to lose its water by evaporation. The material is more resistant to weather than the hot asphalt from which the emulsion is produced. It can be used for moisture proofing concrete, wood, cloth and other materials, and for coating structural steel work, etc. The fire hazards present when dealing with hot asphalt or mixtures of asphalts with solvents are absent when these emulsions are used, and while the latter can be applied to wet surfaces, dry surfaces are essential for the application of the former. At ordinary temperatures most asphalts experience plastic flow, and to overcome this tendency high melting point asphalts must be used. These lack the high ductility and excellent weathering qualities of the lower melting point asphalts. On the other hand, when a soft asphalt is emulsified, the emulsion film is very ductile and will resist flow under heat to a high degree. The property of inertness to flow is due to the formation in a dried film of a "gel" or honeycomb structure through the wall of which the asphalt particles have coalesced on drying. The emulsions may be made in consistencies suitable for different types of application such as spray, trowel, brush, rag, dip and mould. W. H. T.

611. Chemistry of Bituminous Highway Construction: Native Asphalts. J. S. Miller, Jun. *J.S.C.I.* 1929. 48, 782.

The article commences with a brief review of the source and refining processes of Trinidad and Bermudez native asphalts. As obtained, Trinidad asphalt contains roughly 39 per cent. of CS_2 soluble bitumen, 29 per cent. of water and 32 per cent. of mineral and other matter, while Bermudez asphalt averages 68–72 per cent. bitumen, 25 per cent. water and 1–3 per cent. mineral matter. The refining in each case consists of dehydrating in open topped, steam heated stills at about 350° F. The refined asphalts have a penetration of 2 for Trinidad and 20 for Bermudez, and are sold without further treatment or are fluxed with petroleum residuum to form asphalt cement.

Richardson's theory of the formation of Trinidad Lake asphalt is quoted Petroleum of a highly asphaltic nature, rising toward the surface, meets the paste of fine silica and clay which forms the mud of the spring. Emulsification takes place probably owing to the natural gas which accompanies the petroleum at very high pressure. The asphalt thus formed is in a soft condition, but hardens in the course of a year or two. The cause of this and of the gas evolution which accompanies it may be due to the presence of a catalyst, possibly ferrous sulphate, which is known to be present in the clay.

Oxygen and sulphur act similarly with regard to asphalt, and the same general physical characteristics can be imparted to an asphaltic material by air blowing or by heat treatment in the presence of sulphur, both resulting in a marked increase in softening point and a lowering of the ductility.

Organic acids or acid salts are present in all native asphalts, which also contain a certain amount of saponifiable oils, but there seems few, if any, conclusions bearing on the bituminous highway construction problem that can be drawn from their presence or their varying quantity.

While the chemistry of asphalts and its bearing on bituminous highway construction is of vital interest, the material is so complex that little progress has been made. Physical tests, to determine whether certain physical properties indicate suitability, in its broadest aspect, have to be relied on.

Were tests known for determining cohesiveness and adhesiveness little more would be necessary, but in the absence of these methods, tests which differentiate asphalts of different origin and service tests, which prove their suitability or otherwise, have to be used. W. L.

612. Demand for White Oil Introduces New Manufacturing Methods. *The Refiner.* 1929. **8**, (8,) 77–88.

White oils which are stable to light and heat are not easy to make, odour is a vital specification also, and unless an oil remains absolutely odourless for an indefinite length of time, it is not a good oil.

White oils receive the most stringent treatment of any oils, and in the preparation of distillates for white oils, many important points must be considered. It is very important to start with the best grade of crude oil for the purpose, and it would appear that pure paraffin base oils should yield the best distillates, but as a matter of fact excellent distillates can be obtained from purely asphaltic base crudes such as Columbian crude. In the primary distillation, the light oil cuts are taken off and the white oil stock is collected between 39° and 33° Bé., representing about 14 per cent. of the crude oil charged. It shows the following constants: Sp. Gr. 36° Bé., and cloud test 70° F. The latter test indicates the presence of a considerable amount of wax, and before acid treatment of the stock the wax is removed by refrigeration and pressing. It should be carefully processed in order to obtain the best quality of white oil stock. An important point is to chill slowly prior to the formation of wax crystals. Better crystallization takes place if an activator is added prior to chilling, as the crystals build up more uniformly and are purer. The mixture of oil and wax should be brought to a temperature of 14° to 16° F. before being pumped through the presses. The maximum pressure used on the filter press plates is 950 lbs., and it is essential to have a tight joint between the rings and plates to prevent leakage.

The approximate yield of pressed distillate is about 92 per cent. The oil is redistilled with steam, and during this part of the process every precaution must be taken to prevent cracking or ruining the quality of the oil. The best results are obtained when the temperature of the ingoing steam is just a little higher than the boiling liquid. It is detrimental to use superheated steam at 500° F. when the still is only about 320° F. The lighter fractions are

distilled off and the white oil fraction collected. The Sp. Gr. of this fraction is about 28 Bé., Fl. Pt. 345° F. and cloud test 28° F. The yield is about 77 per cent. of the charge. The redistilled stock is treated with 2 per cent. of H_2SO_4 (93·5 per cent.) at 60° to 65° F. for 45 min. At the end of this time, the contents of the agitator are allowed to settle for 5 to 6 hours and the sludge drawn off. The second acid treatment—4 per cent. H_2SO_4—is given in a like manner and the sludge removed. The third treatment is with 98 per cent. acid to the extent of 2 per cent. by weight of the oil charged. Two hours agitation is followed by 12 hours settling and the sludge again removed. The fourth and last acid wash amounts to 4 per cent. of the oil with 2 hours agitation and 12 hours settling as before.

Soda ash is used for neutralizing after the oil has been transferred to the soda washer. After settling for 5 to 7 hours, the soda soaps are removed and the oil is tested to ensure the acidity has been neutralized. When indications show that the oil will wash without emulsifying, steam heat is applied until the temperature reaches 160° to 170° F. and heating is continued for 45 minutes longer. About 20 per cent. of hot water is then added and steam continued for an hour. Agitation is stopped and settling completed in from 6 to 8 hours. The oil is then pumped to bleachers to be brightened.

The final step in the production of white oil is clay filtration, and the oil is charged to the filter by a pump. The head of pressure thus maintained causes the oil to percolate through the clay. The oil is allowed to run as long as the colour is + 20 Saybolt or better. The tests of the finished oil are as follows :—

Colour + 20 Saybolt ; Sp. Gr. 30·5° Bé. ; Saybolt viscosity at 100° F. 76 ; flash 350° F. ; iodine number by addition 0·73, by substitution 14·5 ; British sludge test 1·147 and 1·156. On exposure to light for 30 days no apparent change in colour is observed. The oils are odourless and remain so. Although the above process seems long and complicated, it is actually producing on a commercial basis. R. P.

613. Treatment of Substances Containing Unsaturated Carbon Compounds for the Purpose of Modifying the Physical Properties Thereof. L. Auer. E.P. (i.) 289,414 ; (ii.) 289,415. April 26, 1928. Conv. (Hungary). April 26, 1927.

The process by which unsaturated carbon compounds are modified as to their physical properties by treatment under heat with a metal, a metal oxide or hydroxide, or an organic base is improved by the after-treatment of the modified substance with a material capable of supplying sulphur. This treatment may be assisted by the addition of an accelerator, and the dissemination of the sulphurising agent may be ensured by using a solvent. Two examples are given : (1) 200 Parts castor oil and 4 parts metallic calcium are boiled under reduced pressure until the metal is dissolved and hydrogen ceases to be evolved. After cooling, the mass is heated to 140° C. and mixed with 200 parts iron oxide, 4 parts aniline and 25 parts sulphur. The whole is then heated to 160° C. for ½ hour and the product pressed into moulds. (2) 200 Parts by weight of resin and 10 parts stannic oxide are melted, and after absorption of the stannic oxide are left to cool to 50° C. 20 Parts of petrol and 20 parts of sulphur chloride are added, and after half-hour standing the mass is dissolved in a mixture of turpentine and petrol, the result being a varnish-like substance.

In the second patent, thermoplastic substances containing unsaturated carbon compounds are produced by treating the starting material with a solution of one or more electrolytes in an organic solvent. The electrolytes may be organic or inorganic acids, their salts or esters, metallic derivatives

of organic compounds, oxides or hydroxides of metals or organic bases. The products may be used in the manufacture of varnishes, linoleum, rubber, soap, etc., and suitable fillers, pigments, solvents, etc., may be incorporated.
G. S.

614. Process for the Manufacture of Emulsifying and Stabilising Agents and Dispersions Obtained Therewith. De Bataafsche Petroleum Maatschappij and H. Limburg. E.P. 291,393. May 7, 1928. Conv. (Netherlands), June 1, 1927.

Sulphonic acids for use as emulsifying and stabilising agents are made by repeatedly treating mineral oils or their fractions with quantities, such as 10 per cent. of their weight, of conc. H_2SO_4 or oleum. The treatment may be carried out at raised temperatures, and the sludge removed after each operation until the sludge contains sulphonic acids which are over 250 in molecular weight, are soluble in water and insoluble in benzene, and their calcium salts are soluble in water and in calcium chloride solutions. The acids are recovered from the sludge as such or as their salts.

The sulphonic acids produced can be used for the manufacture of aqueous dispersions of mineral or vegetable oils, tar, asphalt, paraffin, sulphur, rubber, etc.
G. S.

615. Apparatus for Oxidising Hydrocarbon Oils. J. C. Black and W. D. Rial, Assrs. to Pan American Petroleum Co. June 25, 1929. Appl., Nov. 23, 1926. U.S.P. 1,718,679.

The object of the invention is to provide an apparatus by which heavy petroleum residues may be converted into an asphaltic product which will have a high melting point and viscosity, and at the same time be of suitable ductility. The various characteristics of ductility, high melting point and viscosity required by different users may be controllably imparted to the heavy oil separately or in combination. The invention consists of a container for the residue and means for blowing the product with air, superheated steam, etc., in such a manner that the mass of asphaltic material within the container is maintained in a circulating motion and is thus kept uniform.
W. H. T.

616. Treatment of Lamp Blacks and Other Carbon Blacks, and their Industrial Applications. Compagnie Lorraine de Charbons pour l'Electricité. E.P. 300,610. June 8, 1929. Appl., Oct. 31, 1928.

A method of treating lamp blacks, etc., is described, in which the black is incorporated with up to 10 per cent. by weight of tar, natural asphalt or pine tar, whereby the dusty condition is reduced while retaining the pulverulent condition. The tar or other tarry materials are incorporated by grinding, rolling or calendering.
W. H. T.

617. Method of Manufacturing Bituminous Emulsions. G. C. Hurrel. E.P. 315,495. July 18, 1929. Appl., April 25, 1928.

Emulsions of the type in which bitumen is the disperse phase have been made and rendered stable by the use of clays, with the assistance of a colloid mill. A great amount of wear, however, takes place in these mills due to minute crystalline siliceous particles. On the other hand, if paddle mixing is used, the degree of dispersion is not sufficiently great. By means of this invention, the high degree of dispersion obtainable in soap emulsions is secured without subjecting the emulsifying plant to the abraisive action of the clay. A bituminous emulsion is first produced which is unstable to the action of electrolytes and which is characterised by the use of a small proportion of protective colloid. This emulsion is produced in a high speed colloid mill, and is finally added to a suspension of colloidal clay.
W. H. T.

618. Manufacture of Vulcanized Products from Oil. Imperial Chemical Industries, Ltd., and W. J. S. Naunton. E.P. 313,252. June 13, 1929. Appl., April 5, 1928.

Fatty oils consisting of or containing a substantial proportion of unsaturated oils are emulsified with water or other non-solvent liquid with the aid of a suitable emulsifying agent, with or without the addition of a protective colloid and/or alkali. The emulsion so obtained is then vulcanised at temperatures above 100° C. by means of sulphur or a sulphurising agent. Normally this vulcanising is carried out in an autoclave at 160 to 170° C., but by the addition of a suitable organic accelerator, the temperature may be so lowered that a closed vessel is unnecessary. W. L.

619. Production of Rubber (from Buta-diene and Homologues). J. Y. Johnson (Comm. from I. G. Farb. Akt.-Ges.) E.P. 313,188. Feb. 9, 1928.

Buta-diene and its homologues are polymerised to give varieties of rubber in the presence of rubber latex or analogous substances. The latex of gutta-percha or rubber latex known under the trade mark of Revertex may be used. Quantities of 10 to 25 per cent. are often sufficient. The rubber obtained is, after vulcanisation, of good strength. The polymerisation may be effected in the presence or absence of oxidising, emulsifying or dispersive agents or aqueous solutions of the same and may be conducted either in the warm or cold. Ten parts of latex containing 25 to 30 per cent. of caoutchouc hydrocarbons and 5 parts of a 3 per cent. solution of hydrogen per oxide are added to 100 parts of buta-diene and the agitated mixture heated to 75° C. in a closed vessel. In a short time a product separates which can be converted into a valuable rubber by vulcanisation. The higher the proportion of latex used the greater the resemblance of the product to natural rubber. T. A. S.

620. Purifying Gasoline Solvent. C. Macleod. U.S.P. 1,720,604. July 9, 1929. Appl., Sept. 22, 1927.

This is a method of purifying by distillation a dirty high boiling point dry cleaner's solvent containing soaps and other impurities. The solvent is rapidly vapourised by boiling in a vacuum and subjecting the vapourised solvent to centrifugal action, thereby removing impurities contained in the walls of the bubbles formed in vapourising the solvent and entrained in the vapours passing from the still.

The vapourised solvent is removed from the boiling zone at high velocity, and this velocity is used to induce centrifugal action in a Swartwont centrifugal separator installed between the still and the condenser. W. L.

621. The Manufacture of Emulsions, and Emulsifiers for Producing Same. E. C. R. Marks. E.P. 305,742. Nov. 26, 1927.

This invention relates to the manufacture of emulsifiers and emulsions which may be used in the construction and maintenance of roads. An emulsifier is produced in one continuous process by mixing saponifiable material such as a resin, a vegetable gum, or a fatty acid with milk of starch and a saponifier, and heating the mixture with continual agitation until the starch is completely peptonised, the temperature not being allowed to exceed 96° C. on account of evaporation. The mixture is then ready for dilution and use as an emulsifier for bitumen, tar and similar products. D. G. P.

622. Manufacture of Synthetic Resins. G. T. Morgan and A. A. Drummond. E.P. 315,442. Feb. 13, 1928.

Resins are produced under conditions that give products with reproducible characteristics. A phenol is dissolved in an organic solvent such as alcohol

and a solid polymer of formaldehyde is added along with a catalyst. The mixture is then heated, the time and temperature being dependent on the nature of the product required. If an alkaline catalyst has been used, this is removed by treatment with an acid. The resin may be recovered from the solution or the solution may be used in the preparation of moulding powders. Phenol (94 grms.), paraformaldehyde (60 grms.) and lime (1 grm.) are boiled under a reflux condenser with 100 ccm. alcohol for 7 hours. After acidification, washing and drying the solution is poured into 10 times its volume of benzene, when a syrup is precipitated which becomes hard and insoluble when heated to 110° C. The benzene-alcohol solution contains resin which is less readily converted into the insoluble condition. The precipitation of the resin may be carried out on a pulverent material by adding this to the solution. T. A. S.

See also Abstracts Nos. 555, 558, 565, 566, 573, 603, 624, 625 and 628.

Analysis and Testing.

623. Specific Heats of Petroleum Vapours. Bhalke and Kay. Petr. Div., A.C.S. Meeting, April, 1929.

The specific heats of five petroleum distillates were determined at temperatures from complete vaporisation to 650° F. The method used consisted of passing the superheated vapours at a constant rate through a calorimeter to which a known quantity of electrical energy was supplied to be converted into heat, the rise in temperature of the vapours being noted. The oils which did not vaporise at the desired temperature were distilled with steam, and the specific heat of the mixture was corrected for that of steam in order to obtain the specific heat of the oil. The apparatus used included feed tank with steam pump, pipe still, calorimeter with hot air jacket, water cooled condenser, thermocouples and electrical equipment. It was possible using the small pipe still heated by gas to keep the temperature of the vapours constant to within 0·2° F. for over an hour when steam was not used, while with steam the variation did not exceed 0·6° F. The heat supplied to the calorimeter through the chromel resistance-wire coil could also be regulated very closely by the aid of a rheostat. The thermocouples in the calorimeter were of No. 24 gauge iron-constantan wire. Those for measuring the rise of temperature of the vapours were multiple, and so arranged that the voltage per degree was multiplied to four times that for a single couple. The initial temperature of the vapours was measured by a single couple. Tests of the apparatus and of the procedure were made by using superheated steam. The known value for the specific heat of steam could be checked to within 0·7 per cent. when the thermocouples were new, but after exposure to petroleum vapours they gave high values. This change was measured and taken into account when the final results were calculated. At the commencement of a determination, the still and calorimeter were brought up to temperature by means of steam. After the latter was turned off and oil pumped into the still, temperature equilibrium was established; the vapour heater was started, and when equilibrium was again reached the vapour temperature, temperature rise, electrical energy expended in the heater, and still temperature were recorded every five minutes for 80 minutes. The vapour passing through the calorimeter was condensed and weighed. The three light oils showed nearly a straight line relationship between specific heat and temperature. For oils which must be distilled with steam, the average deviation was 2·56 and 2·06 per cent. respectively. The specific heat, temperature (° F.) and A.P.I. gravity are related by the equation: $Cp = 0{\cdot}289 + 0{\cdot}0009G_1 + 0{\cdot}0004t$. W. H. T.

624, METHODS OF TESTING BITUMEN. H. Bösenberg. *Petr. Zeit.*, 1929, **25**, 1127-1134.

The A.S.T.M. methods for testing bitumen which have been adopted in England, Holland and Germany are described in detail and diagrams of the various types of apparatus given. These tests include specific gravity, bitumen content (solubility), softening point, penetration, ductility, and stability (loss in weight or loss by evaporation). The last-mentioned is not dealt with in this article.

The international method for determining specific gravities is given. This constant is determined at 25° C. by means of a pyknometer which holds 30 c.cm. The pyknometer is weighed empty (a), and then weighed full of water at 25° C. (b). The desired quantity of bitumen is then placed in the dried pyknometer and kept at 75–100° C. above the softening point in a drying oven. On cooling, the weight of the pyknometer and bitumen is obtained (c). Water at 25° C. is then added to the mark and the pyknometer is left in a vessel of water at 25° C. After drying, the weight (d) of pyknometer, bitumen and water is noted.

Specific gravity of bitumen $=(c-a)/(b-a)-(d-c)$. This was found to be 1·031 for a German pitch.

The specific gravity is determined at 15° C. in Germany, but it can be estimated at another temperature by using the formula $S_2=S_1 \pm K(t_2 - t_1)$ where $K=\cdot00054$. S_2, S_1 are the specific gravities at temperatures $t°_2$, $t°_1$ respectively, of which S_1 is known, and K is positive when t_2 is less than t_1 and negative when t_2 is greater than t_1.

The solubility of bitumen in carbon bisulphide is next described. 2 gms. of the dry pitch are placed in a 150 c.cm. tared Ehlenmayer flask, and 100 c.cm. of chemically pure carbon bisulphide added. The flask is shaken until the bitumen has dissolved, and left for 12 hours in the dark. The clear solution is then transferred to a clean tared flask without disturbing the residue. The contents of the first flask are then treated with a further quantity of CS_2, shaken vigorously, and left in the dark together with the second flask for 12 hours. The solutions from both flasks are then carefully decanted through 3-mms. thick freshly ignited asbestos fibre in a weighed Gooch crucible. The temperature of the liquid is maintained at 20–25° C. The residue in the filter is washed with CS_2 until the filtrate is colourless, the residues in both flasks are shaken with fresh CS_2 and left for 24 hours, or until the insoluble particles have completely settled. The liquid is then decanted through the filter, and finally the residue in both flasks washed until the washing liquid is practically colourless. The Gooch crucible is kept at 105° C. for half an hour and then weighed. The difference between the weights of the original dry solid and the residue gives the quantity of solid soluble in CS_2. The solubilities of pitch in carbon tetrachloride, choloroform, aromatic-free benzine can also be determined.

Two softening-point methods are given, the ring-and-ball method (A.S.T.M.), which is generally used, and the Krämer-Sarnow method. The accuracy of the ring-and-ball method is within $\pm 0{\cdot}5°$ C., and the results are usually greater than those obtained by the Krämer-Sarnow method. The latter test, in which small rods of bitumen are used, is not very practicable since these rods are susceptible to accidents and temperature deviations.

In the penetration test, a needle of definite dimensions under a load of 100 gms. is allowed to penetrate for 5 seconds into a mould of bitumen kept at 25° C. The needles show no friction towards the pitch, but they must be cleaned with benzol and kept in vaseline. Rusty needles must not be used.

The ductility of a bitumen is measured by the length of fibre to which it can be extended at 25° C. when drawn out at a velocity of 5 cm./minute without breaking. Diagrams of a ductilometer and special moulds for pitches are given. W. S. C.

625. Testing and Analytical Methods Mark Progress in Refinery Technology. H. L. Kauffman. *Refiner*, 1929, **8** (8), 92.

This article discusses some of the analytical methods that have been developed during the past year or two.

Marcusson determines asphaltenes in mineral oils by dissolving 5 gms. of oil in 50 c.c. of ethyl ether and adding 5 c.c. of a 5 per cent. solution of iron chloride. After 2 hours the precipitate is filtered and washed once with ether. The dry filter is then completely extracted with ether. The iron double compound is dissolved in chloroform and washed with 5 c.c. dilute Hce followed by 5 c.c. water, separated, evaporated to dryness and the asphalt dried at 105° C. and weighed.

The method is quantitative and simple, but does not give the same values as the benzene precipitation number.

The asphaltic index of transformer oils is determined by heating 50 gms. of the oil in a 200 c.c. flask to 120° C. for 24 hours in the presence of 10 gms. of pumice fragments previously impregnated with a 7 per cent. NaOH solution and dried. After cooling, the oil is reheated on a water bath for 20 minutes with 50 c.c. of Kisslings reagent, shaken for 5 minutes, the alcoholic solution separated and the asphaltic acids extracted with benzine.

Bukton proposes to quicken oxidation tests by using an oil bath heated autoclave and passing oxygen under pressure through the oil; 6 c.c. samples of oil in uniform diameter glass tubes are used, and the gas at 15 kilograms pressure is circulated at 5 litres per hour for a maximum of 2 hours, the temperature being 150° C.

Nametkin and Abakumovsky define the oxygen number as the active oxygen necessary to oxidise 100 gms. of oil by means of benzoyl hydrogen peroxide.

The number is a measure of the unsaturated hydrocarbon content. 0·5 N solution of benzoyl hydrogen peroxide is used, at a temperature of 9 to 12° C. in the dark for from 40 to 48 hours.

Kattwinkel proposes a method for determining aromatic and unsaturated hydrocarbons. 10 c.c. of gasoline is treated with 30 c.c. of a solution of 30 gms. phosphorous pentoxide in 100 c.c. of 1·84 sulphuric acid. The mixture is shaken for 5 minutes. Increase in acid volume gives aromatic and unsaturated content. If a solution of 20 gms. boric acid in 100 c.c. of 1·84 sulphuric acid is used in place of the phosphorous solution, the unsaturated content alone is obtained.

Dietrich defines spirit value as the critical solution temperature of seven parts by weight of gasoline and three parts by weight of 96 per cent. alcohol. He finds a measure of inter-relation between spirit value and resistance to knocking, and also that gasolines having a very high unsaturated content show good behaviour with regard to knock resistance.

Stevenson and Babor determine the volatility of gasoline by means of the "black-surface dew indicator," whereby determinations are made easily, rapidly and reliably.

Mallison has developed a stability test for bituminous emulsions. The water content is determined and the bitumen found by difference. 10 gms. of the emulsion is spread over an area of 100 sq. cm. on a glass plate, and fine sand (5 gms. per gm. of bitumen) is spread over it. The plate is weighed and allowed to stand at room temperature, being weighed at

intervals until 50 to 80 per cent. of the water has been lost. The external condition and softness of the layer are noted and the plates placed at an angle of 45° in water at 20° C. for 24 hours. If the bituminous layer still adheres firmly it is placed in a level position under a vertical spray of water for 20 minutes.

In Young's direct oil-corrosion test warm oil is run continuously over warm steel, white metal, brass or any other metal, the temperature employed being 90° C. In connection with this he devised a sulphate value test. 20 c.c. of potassium hydroxide solution (210 gms. in 300 c.c. water) is added to 100 c.c. of sample and kept just below 100° C. for 3 hours with air bubbling through. 90 c.c. of hot water is added and the whole shaken thoroughly. 70 c.c. of the alkaline solution is tapped off and the sulphate precipitated with barium chloride in the usual manner.

Graham estimates unsulphonated residue in spray oils by gradual admixture with 38N sulphuric acid (temperature kept below 60° C.) heating to 60-65° C. for 10 minutes, and shaking for periods of 20 seconds at 2-minute intervals. The unsulphonated residue is measured after centrifuging for 5 minutes at 1200–1500 revolutions per minute.

Kennedy describes a method for determining sulphur in volatile fuels. The fuel is vaporised in a carburetting device and burned, the sulphur oxides being absorbed in sodium carbonate solution of known strength which is titrated at the conclusion of the test. The method is said to be precise and rapid.

McKee and Parker state that 25 per cent. acetic acid is the best reagent to use for the rapid determination of nitrogen bases in petroleum distillate.

Whitaker determines carbon and hydrogen by ignition in a Couthal bomb under high pressure of oxygen. The water formed is collected in calcium chloride placed in the bomb and in a U-tube of sulphuric acid on pumice. The discharged gases are passed through a meter, and after measuring, the carbon dioxide is estimated in a Haldane apparatus.

Wik has found that carbon in most aliphatic and aromatic compounds oxidises to carbon dioxide and acetic acid in potassium dichromate solution if the compound is soluble in water or acetic acid.

Berl, Schmidt and Winnacker describe a method for estimating carbon and hydrogen in which lead chromate is used when burning the substance for analysis.

Faragher, Morrell and Monroe give a procedure for the quantitative determination of sulphur present in naphtha as hydrogen sulphide, elementary sulphur, mercaptans, sulphides, and di-sulphides.

The determination of ketones in petroleum presents great difficulties. The sodium-bisulphite, sulphuric acid, water extraction, phenyl hydrazine methods all either give uncertain results or else are applicable only to certain ketones. W. L.

626. Lubricating Oils: Carbon Residue Estimation. The Ramsbottom Method. C. I. Kelly. *J. Inst. Petr. Techn.*, 1929, **15**, 495–515.

Repeated endeavours have been made to devise some means for selecting out of a number of lubricating oils the one which would give the least "carbon" when in actual use in internal combustion and other engines. To this end, various investigators have either developed methods or have made improvements in existing methods. The author gives a historical review of the development of the Conradson carbon residue test which culminated in the A.S.T.M. Standard Test D.181-27T of 1928. Endeavours to arrive at an accurate method of estimating carbon residue have also led to the development of the Ramsbottom test, which has been adopted as

a standard method by the I.P.T. (Serial Designation G.O.9a). Two types of apparatus are described, both satisfying the details of the method of test, but only one being satisfactory in actual use. In the first apparatus heating was difficult owing to excessive radiation, manipulation was not easy and satisfactory temperature control was only obtained by using an automatic gas pressure control. The main objectionable feature, however, was the risk, when heating rapidly from cold, of the formation of small fountains of solder and subsequent entry of the latter into the metal sheaths. Among other objectionable features were the difficulty of placing the glass coking bulbs in the sheaths, the formation of excessive dross on the surface of the lead, brittleness of the cast iron container at 550° C. and non-uniformity of temperature. In order to remove these objections, the second apparatus was devised which has given satisfactory working. This consisted of five parts:

1. A gas ring burner fitted with manometer and needle valve control.
2. A sheet metal container for the molten lead bath.
3. A circular lid to the under side of which were welded the metal sheaths, holes being drilled in the lid to permit of entry of the coking bulbs into the sheaths. An additional hole was made for accommodating the pyrometer, and from the under side of the lid was hung a cylindrical skirt of sheet metal on the inside of which was hung a layer of asbestos.
4. A circular base plate with a circular hole. On the upper side of the base plate a cylinder was welded of such a size that it was an easy sliding fit into the cylindrical skirt of the lid.
5. A vertical rod passing through the upper cylinder and resting in a ferrule welded to the base plate. The rod had a circular eye welded to the top so that the pyrometer could be rested and kept in an inclined position.

The apparatus was thus well insulated, radiation was at a minimum and temperature control was fairly simple. Lead could not enter the iron sheaths, dross formation was largely prevented and, owing to the construction of the apparatus, convection currents in the molten lead were nearly eliminated. In order to ascertain the effects of the variables, a number of estimations were carried out in batches of six at a time. Firstly, it was found that there was very little difference between the behaviour of the coking bulbs in the six sheaths, and that several sets of six determinations could be carried out in quick succession without the results obtained being affected by temperature variations of the sheaths.

Secondly, the greatest carbon residue was obtained when the final heating period was least, but the percentage difference was greatest on oils of low carbon residue. Again, the carbon residue was higher, the smaller the weight of oil taken, but variations in the size and thickness of the bulbs had no effect. The lower the temperature, the higher the result, due probably to the varying time factor. It is considered that, using the improved apparatus, different laboratories using the same oil should agree within much less than the 10 per cent. tolerance. W. H. T.

627. A Method of Colour Determination in Mineral Oil Refineries. H. Magnus. *Petr. Zeit.*, 1929. **25**, 984-986.

The apparatus described is Stamnaer and Saybolt's colourimeter. The total horizontal length is 68 cms. and its breadth is 17 cms. On the near side which faces the source of light is a glass porcelain disc which can be removed from the apparatus. The front part is protected from side lights by means of a black metallic plate. The bridge in the centre which covers the eyepiece can be removed so as to clean and fill the apparatus.

T

The essential improvement of this apparatus compared with the known models is its portability, for it can be operated on any table or other type of support. This is attained by an ingenious eyepiece which totally reflects the light rays at an angle of 90 deg. above the observer's eyes; thus it is possible to use horizontal glass tubes or troughs about 40 mm. deep with a "colourimetric" length of 300 mms., in place of very deep tubes of liquid. By means of a deflection eyepiece which can deflect the image at 60 deg. the colour determination can be conducted in a sitting position. The procedure is as follows: One glass trough is filled with a standard solution and the other contains the solution under test, or a test glass, *e.g.*, an uranium glass disc may be utilised in place of the standard solution. In the latter case the trough on the standard side is filled with water in order to obtain equal illuminating capacities. Both troughs are illuminated from the glass porcelain disc by day or lamplight. After the light rays have penetrated the liquid under test, they strike the immersed prism which throws the incident light by total reflection upwards into the eyepiece. There are now two adjacent fields of colour in the eyepiece, and by slowly horizontally displacing the trough containing the test solution until the colours are identical, the readings in centimetres and millimetres are read on the lower scale and the relative degree of brightness of the sample is finally calculated from the formula :—

$$K_2 = K_1 . H_1 / H_2$$

where

K_1 is the known colour value of the standard solution or of the test-glass.
K_2 is the desired colour value.
H_2 is the depth of the layer of the liquid under test.
H_1 is the depth of the layer of the standard solution.

When using a standard solution it is advisable to keep some at hand in closed tubes in order to protect it from the oxidising influence of the air. The use of a test-glass in lieu of the standard solution enables one with quick handling of the apparatus to revert from the testing of benzine to any desired fraction as far as the lightest machine oils.

The apparatus can be used in the laboratory and in distillation or refinery plants as a continuous means of control of the homogenity of the refined products from benzine to spindle oil fractions. Refined products with a viscosity greater than 2·6° at 20°C. can be determined colorimetrically with this apparatus since they have a sufficiently strong individual colour. It can also be utilised to determine the colour values of paraffin, Montan wax, and similar bodies of low-melting point. For this purpose the instrument is mounted on an iron plate and fitted with a heating apparatus which can heat the base of the instrument to any definite temperature.

The "Hellige-Block Comparator" is also mentioned. This can be carried in a pocket and is used for the colour testing of refined products, also of acids, lyes, boiler feed water, waste water, and for determining P_H values.

W. S. C.

628. THE ESTIMATION OF THE SOFTENING POINT OF PITCHES AND ASPHALTS. E. Spilker. *Petr. Zeit.*, 1929, XXV, **25** (835-836).

A method of determining the softening point of pitch, the Kraemer-Spilker method is described and recommended. The dropping body consists of a small smooth rod of type metal (80/20 parts of lead and antimony) whose diameter is 5 mms. and whose lower end is hemispherical. Slight deviations from the standard weight are found to have very little effect on the softening point, and the results obtained are comparable with those obtained by the Kraemer-Sarnov method in which the dropping body is a small rod of mercury weighing 5 gms.

W. S. C.

629. METHODS OF QUANTITATIVE DETERMINATION OF AROMATIC HYDROCARBONS IN GASOLINES PRODUCED BY STRAIGHT DISTILLATION. M. D. Tilitscheyew and A. I. Dumskaya. *J. Inst. Petr. Techn.*, 1929, **15**, 465-483.

The formula generally used for the determination of aromatics is

$$x = k \cdot d,$$

x being the percentage of aromatics by weight, k a coefficient obtained by experiment, and d the difference between constants before and after separation of the aromatics. The constants chiefly used are the C.S.T. of the gasoline in various liquids (aniline, nitrobenzene, etc.), the specific gravity and the refractive index.

The four methods, aniline, nitrobenzene, specific gravity and refractive index have been investigated, and for the benzene, toluene and xylene fractions the refractometric method seems to be the best. Only small quantities of the gasoline are required, results are accurate to $\pm$ 0·3 per cent., and the time needed is only about two minutes, as compared with at least 15 minutes for an aniline point determination.

Where greater accuracy is required for fractions boiling below 150°, the specific gravity method is almost as accurate as the C.S.T. methods.

Aromatics in the 150-200° position should be determined by the aniline or nitrobenzene method, as the specific gravity and refractive index methods can only be successfully applied to fractions boiling below 150°.

The nitrobenzene method is generally preferred to the aniline owing to it being more rapid, due to the lower C.S.T., the lesser fluctuation of nitrobenzene coefficients, and the greater stability of nitrobenzene.

It must be noted that the volume ratio of aniline to petroleum fraction depends on the percentage of aromatics. For an aromatic-free gasoline, 0·6 to 0·7 vol. per 1 vol. of fraction is necessary, while for a fraction containing 10 per cent. aromatics 0·9 to 1·0 vol. is required. G. S.

630. A MODIFICATION OF THE LAMP METHOD FOR THE DETERMINATION OF TOTAL SULPHUR IN PETROLEUM DISTILLATES. Wood and Mattox. *Petr. Sec.*, A.C.S. meeting, April, 1929.

The difficulty in determining total sulphur in the lightest fractions of petroleum by either the lamp or the bomb method has led to an improvement in the former. Two strands of cotton wick are passed through a glass tube 12 cms. long and 4 mm. inside diameter, the latter being inserted in a cork which fitted into a test tube graduated in cubic centimetres. About 15 c.c.s. of the gasoline to be tested is placed in the test tube, the cork and wick are attached and the tube placed in ice water, the level of which could be regulated by the aid of a levelling bulb. With very light gasoline, a small steady flame can be maintained by keeping the ice water level about half an inch above that of the sample. With grade A natural gasoline, the level of the ice water is about the same distance below that of the gasoline. After all adjustments are made, and the apparatus is ready, burning is continued for 90 minutes, after which the quantity of sample remaining is read. With ordinary gasoline and with kerosines, the ice water bath is not used, while with crudes and heavy distillates the ice water is replaced with a heated liquid bath and adjustments made as before. Good results are experienced if the oil does not contain asphaltic or tarry matter. W. H. T.

631. VOLUMETRIC ESTIMATION OF SULPHUR ON CRUDE PETROLEUM. G. Woodward. *Ind. and Eng. Chem., Anal. Ed.* 1-iii-117.

Nikaido has suggested the estimation of sulphate in 50-70 per cent. alcohol solution by titration with lead nitrate using potassium iodide as indicator, since in this solution lead iodide is not permanently formed until a quantitative

precipitation of lead sulphate is obtained. Where the amount of sulphuric acid is 0·20 gms. the error in endpoint does not exceed 0·4 per cent., while with 0·03 the error does not exceed 3·0 per cent. The solutions suggested are 33 gms. lead nitrate made up to 1 litre with water and standardised against sulphuric acid made by the addition of 50 c.c.s of water to 20 c.c.s sulphuric acid, which is then added to 100 c.c.s of 95 per cent. alcohol.

In the actual estimation, the crude oil is burned in an oxygen bomb containing water and the aqueous sulphuric acid, if yellow due to iron salts, is boiled with a small quantity of powdered aluminium until colourless. Alcohol is added in sufficient quantity to make the solution 50-70 per cent. alcohol and the titration carried out in blue light till the lead iodide yellow develops. A. R. S.

See also Abstracts Nos. 576, 577, 578, 584, 586, 587, 589, 600, 642 and 654.

Refinery Plant.

632. Handling Sulphurous Crude in Refineries. D. G. Cooper. *Oil and Gas J.*, 1929, 27 (48), 164, 177-178.

The dangers attached to the handling of sulphurous crudes are so general that they present a problem which requires systematic and scientific handling. Hydrogen sulphide, which is in solution in the oil or is formed during distillation, is extremely poisonous and also very corrosive to some metals.

In dealing with the problem, the ideas of all departments must be co-ordinated in order that the rules and regulations for safety are as complete as possible. In the first place the crude must be investigated in the laboratory so that the chemical aspects can be examined and the various operating units informed of the risks to be encountered. The safety department must then draw up rules and regulations for the safety measures to be adopted, which must be strictly adhered to in all branches of the operations. G. S.

633. Designing Flash Distillation Equipment for Petroleum Refining. R. S. Peroomov and N. E. Loomis. *Chem. & Met. Eng.*, 1929, 36, 472.

The term "flash distillation" applies to any operation in which a mixture of hydrocarbons, after being heated above its initial boiling point, is discharged into a zone where separation of vapours from residual liquid can occur. Distillation occurs under conditions of substantial equilibrium between vapour and liquid in the separating zone. The equilibrium single-flash curve of a stock starts at a higher and ends at a lower temperature than either the true boiling point or the Saybolt 10 per cent. distillation curves, crossing the last two somewhere in the lower middle range. It is possible to establish a simple relationship for the slopes and points of intersection of flash curves with the slopes and mean boiling points of the corresponding true boiling point or Saybolt 10 per cent. distillation curves. This relation holds only for the distillation curve at 760 mms. If the true boiling point distillation of a stock is available, the point of intersection and the slope of the flash curve may be read off and the straight line drawn to represent it will approximate very closely to the actual equilibrium curve at atmospheric pressure. Experimental results indicate that the slope of the flash curve and the percentage off at its intersection with the true boiling point curve are practically independent of pressure. Therefore, all that is necessary to estimate the flash curve of a stock, at any other pressure than 760 mms. is to extrapolate the temperature at the intersection of the two curves at 760 mms. to the desired

pressure by using the Cox chart for vapour pressure of hydrocarbons. Concrete examples are given showing how steam consumption, and the temperatures at top and bottom of the bubble tower, etc., can be obtained from these curves.

W. L.

634. White Star Operating Detroit Plant. C. O. Willson. *Oil and Gas J.*, 1929, 28 (10), 62, 229.

The White Star Refining Co. is now operating its 6000-bbl. refinery at Trenton, seventeen miles from Detroit. In common with all the new larger plants, the consuming market has determined the location.

Erection work started on February 1, 1929, and the plant, the whole of which is new, was ready to operate the first week in July. The plant consists of two 3000-bbl. distillation units, two 1500-bbl. Dubbs cracking units, a 3300-bbl. re-run still, and necessary auxiliary plant, including a power plant.

The distillation units may be operated to yield gasoline, naphtha, kerosine, distillate and gas oil as overheads. The crude, preheated to 250–275° F. is discharged into a dehydrator and then fed direct to the tube still for fractionation, the vapours passing to the tower and the residuum removed to storage. Gasoline is taken off as the main overhead stream from the tower, the second stream being kerosine and the third gas oil. Two 4 × 14-ft. flash towers are used in the final fractionation.

The re-run still is designed to treat the Dubb's preasure distillate, the gasoline vapours being passed to two Gray towers for further refining in the vapour stage. Two flash towers supplement the work of the main tower used with the re-run still.

G. S.

635. Process for Thermally Decomposing Hydrocarbons. Electro Metallurgical Company and S. M. Norwood. E.P. 298,556. July 25, 1929. Appl., June 19, 1928.

It has already been proposed to conduct the cracking of hydrocarbons at 900–1000° C. in the presence of an iron-chromium alloy, a preferred composition containing upwards of 20 per cent. of chromium. This alloy is workable, highly resistant to heat and to oxidation, and in contradistinction to iron, it is practically devoid of that type of catalytic effect upon hydrocarbon vapours which leads to serious deposition of carbon. Unfortunately, a characteristic brittleness is developed in iron-chromium alloys containing 20–35 per cent. of chromium when they are held for some time at 400–500° C. as may occur in slowly cooling a cracking apparatus; furthermore, fusion-welding of such alloys or others of even low chromium content, often produces a zone of brittleness near the weld.

It is found that the embrittling action of temperatures of 400–500° C. may be inhibited by the addition of 2–15 per cent. of nickel, the tensile strength and other mechanical properties of the alloy being also improved. The nickel tends, however, to make the alloy more readily corroded by sulphur or sulphurous gases. This tendency may be overcome by a suitable addition of silicon with the nickel. The silicon, especially in conjunction with a suitable proportion of manganese, also obviates the brittleness produced by fusion-welding. The alloy preferred contains, therefore, 15–40 per cent. of chromium, 2–15 per cent. of nickel, 0·7–3 per cent. of silicon and 0·7–3 per cent. of manganese, the balance being principally iron. Such an alloy is readily forged, rolled, etc., if the carbon content is not greater than 1·0 per cent.; it preferably should not exceed 0·30 per cent.

W. S. N.

636. Separating Oil and Oil Vapours. J. Primrose, Assr. to Foster Wheeler Corporation. U.S.P. 1,723,748. Aug. 6, 1929. Appl. May 8, 1923.

The oil and vapour mixture is supplied under some pressure to a distributor centrally disposed in a vertical lagged cylindrical separating tower. The distributor causes flat radial jets to impinge on the walls at high velocity, thereby effecting an initial separation of oil and vapour. The oil flows downwards in film-like and broken streams over the surface of conical spreaders comprised of sectors provided with transverse ribs, and having their apices alternately downward and upward. These baffles are easily assembled in the tower and are also claimed to reduce heat loss from the walls. Superheated steam is admitted from jets around the base of each of the cones having its apex upward, and any vapours liberated below these cones are passed upward from their apices by inclined pipes, thereby avoiding contact with descending oil. Each of the inverted cones makes a gas-tight joint with the wall, vapours being withdrawn from each pocket so formed, and transferred to a point immediately above the inlet. Oil freed from vapour is drawn off from the bottom of the column through a seal pipe.

Vapours ascending from the inlet and those liberated from the baffles are freed from entrained oil by layers of packing material supported on grids in the upper portion of the tower, and finally the vapours are made to pass around a stationary horizontal spiral, liquid being returned on to the top baffle through a seal. L. H. A.

637. Method of Simultaneously separating Mixed Liquids of different Boiling Points and Generating Power. C. F. Hirshfeld. U.S.P. 1,716,130. June 4, 1929. Appl. Mar. 6, 1922.

The mixed liquids are vaporised under pressure and the cooling of the mixed vapours is accomplished by passing them through a multiple expansion power producing apparatus wherein the expansion of the vapours provides the cooling effect for their condensation and at the same time is utilized for the production of useful work.

In the preferred form, the apparatus consists of a multi-stage turbine generator which is connected directly to the vapour line of the still. Each stage of the turbine is provided with a drain and the fractions which are condensed in the different stages are drawn off from these drains. Vapours which pass the turbine without condensation are led into a condenser. By suitable design of the turbine it is possible to provide stages that will condense predetermined fractions. It is claimed that the method is applicable o steam or dry distillation. W. L.

638. Distillation of Hydrocarbon Oils. W. W. Holland, Assr. to Standard Oil Co. of Indiana. U.S.P. 1,723,698. Aug. 6, 1929. Appl. Dec. 11, 1924.

The invention relates to improvements in the distillation of hydrocarbon oils in which the vapours arising from the distillation of oil are subjected to partial fractionation. The partially fractionated vapours are passed in indirect heat conductive contact with a current of steam at a temperature of 220°—240° F., thereby heating the steam and partially condensing the vapours. The condensate thus formed is returned to the still in countercurrent to the ascending vapours, and the steam is passed into direct contact with a distilling body of oil. W. H. T.

639. APPARATUS FOR THE CONVERSION OF HYDROCARBONS. L. C. Huff, Assignor to Universal Oil Products Co. U.S.P. 1,716,136, June 4, 1929. Appl. Feb. 23, 1924.

Heavy oils, such as residuum, are subjected to the direct action of high temperature steam injected into the body of the oil. The steam conduits to the still are positioned immediately below the bottom of the still, between the source of heat and the body of the oil, thus accomplishing the double purpose of superheating the steam to any desired temperature and screening the bottom of the still to prevent the heating medium from impinging directly on the bottom surface.

The interior steam conduits are perforated on their lower sides and spray the steam against the bottom of the still. It is claimed that this gives maximum absorption of heat from the still bottom and that carbon accumulation is avoided, and as the mass is maintained in a liquid condition the danger of hot spots is practically eliminated. A temperature of 800 to 1000° F. may be maintained in the still and the system may be worked from atmospheric pressure up to a pressure of several hundred pounds. W. L.

640. SEPARATING GASOLINE FROM MINERAL OIL. A. E. Pew and H. Thomas' Assrs. to the Sun Oil Co. of Pennsylvania. U.S.P. 1,723,368. Aug. 6, 1929. Appl. Feb. 23, 1924.

A process is described of separating relatively light and relatively heavy gasoline from mineral oil, with a maximum of simplicity and a minimum expenditure of heat. A flowing stream of oil is heated to a temperature above the vaporising point of a relatively light constituent thereof, whereby the oil is partially vaporised. The vapours are fractionated in a space heated by the flowing oil and then removed. The residual oil is passed to a second chamber where it is heated by flowing over an extended surface in a thin layer and subsequently collected in a pool. Heat is applied to the pool and the flowing thin layer of oil so that the hot vapours driven off by this means come into contact with the thin layer of oil. The vapours from the second chamber are removed to a third space wherein fractionation and removal of vapours again takes place. W. H. T.

See also Abstracts Nos. 567, 569, 570, 574, 575, 579, 581, 582, 606, 610, 676, 682, 694 and 695.

Engineering.

641. OPERATING FACTORS AND ENGINE ACCELERATION. D. B. Brooks *S.A.E. Journal*, 1929, **25**, 130–136.

The author describes tests subsequent to those detailed in the *S.A.E. Journal*, June, 1929, and arranged for the study of the effect on acceleration of fuel distribution, jacket water temperature, and spark advance. He also describes deceleration tests and applies these to the measurement of friction under conditions approaching those of normal running more nearly than the usual method of motoring by means of an electric dynamometer.

The six-cylinder engine and the recording apparatus were used as before but, in order to determine distribution effects, the engine was run without electrical load and was accelerated on two cylinders. Prior to each test, the engine was run under standard conditions on all cylinders, four cylinders being switched off during the measured acceleration period. Cylinders 1 and 2, 3 and 4, and 5 and 6 were tested in pairs, and 3 and 4 were found to give the best overall acceleration; of the other pairs one gave better initial acceleration than the other but fell behind in the later stages of the speed rise. There was only very slight variation between standard fuels D, E and F. Tests on

U.S. Motor gasoline with the cylinders paired 1–6, 3–4, 2–5 to give equal firing intervals differed only little from the previous tests, and again the pair including cylinder 2 was poorest, indicating bad distribution to this cylinder. As the manifold temperature was increased the difference between the pairs fell to a negligible amount. Runs on benzole shewed a still closer agreement, especially above 400 R.P.M. These effects were to be expected from the results of the previous work on acceleration of the six cylinders taken together. Other tests had suggested that jacket temperature had little effect on acceleration but the conclusion was based on runs at a manifold temperature of 80°C. Repeat tests were undertaken at a manifold temperature of –7°C. and with jacket temperatures from –19°C. (brine cooled) to 108°C. (steam heated). Making allowance for the lower starting speed of some of these tests the acceleration was found to be substantially the same for jacket temperatures from –19 to 31°C. The air fuel ratio used was about 9 to 1. Above 31° the acceleration was found to *decrease* up to 700 R.P.M. This is believed to be due to the greater evaporation of fuel at the higher jacket temperatures giving an over-rich mixture. It is concluded from this series of tests that with air fuel ratios sufficiently rich to ensure good acceleration at low speeds, poor acceleration is obtained at high speeds when the jacket temperature is high. In addition there is also loss of acceleration at both extremes of jacket temperature, that at the higher being ascribed to lower volumetric efficiency, and that at the lower to the rapid increase of engine friction.

The effect of spark advance on acceleration is generally that which would be expected from the results of brake torque, benzole giving superior results on account of improved volatility but requiring a lower optimum advance. To simulate the effects of low gear conditions the inertia loading disc of the engine was removed and the electric load cut off. In general, rich mixtures then gave better acceleration at the start, but fell short of the leaner runs at the higher speeds.

In order to determine engine friction effects, the retarding forces due to windage, electrical load and dynamometer friction were determined and deducted from the total retarding force calculated from the known total inertia of the system and the deceleration. Values obtained in this way were compared with values from motoring tests made by driving the engine from the dynamometer immediately after each test. At high manifold and jacket temperatures the values found by the two methods were almost identical. At low jacket temperatures the motoring test gave nearly double the friction, but the deceleration test shewed only 20 per cent. to 40 per cent. increase. When the crankcase oil was renewed there was only a small change in the friction measured by deceleration although the motoring method shewed an appreciable increase. In all cases the friction measured by the deceleration was found to increase sharply just as the engine ceased firing, the cause being obscure and still under investigation. It is concluded that engine friction measured by motoring tests does not necessarily represent operating friction due to the motoring test being affected by renewal of lubricant. The friction at a given speed and temperature at full load varies with cylinder wall temperature resulting from the conditions of operation, and at high temperatures the variations are only small. R.S.

642. Relation between Atmospheric Temperatures and Engine Operation with respect to Fuel Volatility. G. G. Brown, *Oil and Gas J.*, **28**, (7), 1929, 46.

The first part of this paper continues the discussion on the relation between the requisite effective volatilities of motor fuels, under various operating

conditions, and the A.S.T.M. distillation curves for the same fuels. Previous papers by the author on these lines appeared in *Oil and Gas J.*, **27**, 42, 1929, 107 and in **28**, (5), 1929, 46.

Further, a report is given of road and laboratory tests made to elucidate the causes of vapour locking. Both sets of tests deal with the measurement of fuel temperatures in the feed line and carburettor bowl under varying atmospheric conditions. The influence of pressure feed is examined and found to be advantageous in preventing locking. A correlation is made between lock tendency and vapour pressures, at a given vapour-liquid ratio determined in the Reid vapour-pressure bomb.

The conclusion is reached that the problem of vapour lock is mainly one of proper design of fuel-system so that the fuel is not overheated before leaving the jet. G. B. M.

643. Study of Oil Sprays for Fuel-Injection Engines by means of High Speed Motion Pictures. E. G. Beardsley. *Oil and Gas Power*, 1928, **50**.

Experiments are described to determine the characteristics of fuel sprays for airless injection engines with particular reference to penetration, general shape, and atomisation when injected into dense air. It is pointed out that the fit of a spray into the combustion head is of some importance in the design of high speed aircraft engines. The apparatus used consists of a bank of condensers coupled to a rotating distributor switch and arranged so that the discharge spark from each is focussed on the oil spray. The condensers are charged through two kenetrons and a transformer to 30,000 volts. The time of discharge is short enough to allow of an exposure of 1/600,000 sec, and the intensity sufficient to give the necessary illumination. The frequency of the discharges is controlled by means of a rotary multiple break type switch. The sprays are produced by means of pressure delivered by a hand pump large enough to maintain pressures up to 12,000 lb. per sq. in. during valve opening, and the timing valve consists of a spring loaded needle valve operated by a cam driven lever. The cut-off is regulated with a second cam driven poppet valve. The spray is directed into a cast iron chamber with optical glass windows in opposite sides. Gas pressures up to 600 lb. per sq. in. are used in the chamber, and records are made on a strip of film wound on a motor driven 30 in. circumference drum arranged in a suitable camera. The following investigations are recorded:—

1. *Effect of gas density on penetration.* The general conclusion is that penetration with a given spray valve depends solely on the density of the gas in the chamber. In the centrifugal sprayer tested, it fell almost in linear relation with the rise of absolute pressure up to ·001 sec. from the start of the spray. At ·002 and ·003 secs. from the start of the spray the penetration fell quickly as the chamber pressure was increased, the rate of fall decreasing as the higher pressures were reached. Pressures up to 950 lb. per sq. in. were examined.

2. *Effect of specific gravity on penetration.* The general conclusion is that penetration increases with gravity of the fuel used.

3. *Effect of groove angle of helix controlling centrifugal action.* The greater the helix angle, the greater is the penetration. A 90° angle gave a non-centrifugal spray. The ratio of spray volume to oil volume was greatest with a centrifugal spray and the ratio was nearly doubled when a 23° helix was used as compared with the 90° angle.

4. *Effect of alteration of ratio of nozzle area to groove area.* The penetration decreases rapidly with a small increase of ratio, but this is followed by a more gradual change and reaches an almost constant value at a ratio of 2 to 1.

These tests also indicated that orifice size has a considerable effect on distribution and atomisation.

5. *Effect of type of spray on distribution.* A centrifugal spray is concentrated with increase of chamber pressure, and a non-centrifugal spray is spread slightly as the chamber pressure increases. Increase of nozzle area increases spread slightly.

A test is recorded in which a secondary discharge was observed; this was thought to be due to a pressure wave effect in the fuel line and was eliminated by increasing the length of fuel inlet pipe between pump and spray valve.

6. *Effect of injection pressure on penetration.* The penetration increases almost directly as the injection pressure although there is high upper limit beyond which further increase may decrease penetration due to excessive atomisation. Increase of injection pressure reduces the spray angle of a non-centrifugal spray and increases that of a centrifugal one. R. S.

644. Oil Spray Investigations of the National Advisory Committee for Aeronautics. W. F. Joachim. *Oil and Gas Power*, 1928, **50** 1–8.

Six major methods of investigating oil sprays are discussed. Injection into air is often misleading when it is applied to determine the shape and size of the spray actually injected into the engine. Injection into liquids, particularly by Woltjen's method, has enabled the spray particle size to be examined. This method involves spraying into a bomb under various air pressures and collecting the oil particles in a solution of 70 per cent. distilled water and 30 per cent. "Quesol D" in the bottom of the bomb. It could be extended in scope and would permit of the practical determination of spray particle size for many test conditions. Injection of sprays on targets on a flywheel gives important records of lag, rate, and duration of injection. Injection into unheated compressed gases has been developed and described by Beardsley, and is also reported on by the author. Injection into heated and compressed air has, in general, been limited to the determination of ignition temperatures and the time lag between injection and ignition. The Advisory Committee for Aeronautics has developed an apparatus for the combined examination of the effects of air pressure, temperature, turbulence, combustion chamber shape, exhaust gas dilution, and all the variables of injection, atomisation and distribution upon the auto-ignition temperature, lag of auto-ignition, and rate of combustion of oil sprays.

Injection into engines is usually limited to engine performance tests, and is not directly applicable to the study and analysis of sprays because of the influence of chamber design and other variables. The method is too costly for fundamental research when properly developed.

The author describes tests made with the N.A.C.A. high speed photographic apparatus and gives curves shewing the penetration from a given nozzle with various injection pressures into chamber air pressures from atmospheric up to 300 lb. per sq. in. The penetration up to 5 in. into atmospheric pressure air is almost linear with time, and increases with injection pressure. It falls as the chamber air pressure increases and is no longer uniform with time, tending to reach a limiting value with low injection and high air pressures. Photographic records of the spread and penetration of centrifugal sprays into pressure chambers indicate that combustion chambers designed after the examination only of spray into atmospheric air may result in inferior engine performance. Work is in progress to determine the factors controlling the occurence and velocity of pressure waves in injection systems for a range of conditions, and also to determine the exact action, injection lag, operating pressures and fuel delivery rates of injection valves. R. S.

645. Efficiencies of Otto and Diesel Engines. F. O. Ellenwood, F. C. Evans and C. T. Chwang. *Oil and Gas Power*, 1928, **50**, 50–55.

The authors consider that the best measure of the performance of an internal-combustion engine is that called by the A.S.M.E. Test Code on Definitions and Values the "Engine Efficiency." This gives a comparison of the brake thermal efficiency of the actual engine with the ideal efficiency of an engine operating on the same fuel-air mixture at the same compression ratio. The paper gives the results of thermal efficiency calculations for ideal engines operating on the Otto and Diesel cycles with a number of different fuel-air mixtures at various compressions. The temperature-specific heat data collated by Goodenough and Felbeck (*Bull.* No. 139, University of Illinois, 1924) were employed. The "engine efficiencies" of several modern engines under varied conditions were evaluated by comparison of the calculations with test results. The ideal Otto cycle is taken as one in which there is no transfer of heat from or to the working substance whilst in the cylinder and no leakage, fluid or mechanical friction. The mixture at the beginning of compression is assumed to be a gas at atmospheric pressure and 200° F., the mixture strength being the same as in the actual engine provided that the latter has at least sufficient air for complete combustion of the fuel. No mixture containing less air is dealt with in the calculations. The authors consider that a deficiency of air should be indicated by a reduced "engine efficiency." The combusion takes place at constant volume. The Diesel cycle necessitates certain modifications in the assumptions. The fuel is eliminated from the mixture during compression, at the end of which it is injected in the liquid form and the subsequent combustion is at constant pressure.

In the calculations, the temperature at the end of compression is first arrived at by the "trial and error" method. The specific heat of the mixture and the compression ratio are known and a definite initial temperature (200° F.) has been assumed. Owing to the variation of the specific heats of the component gases with temperature a temperature must first be "guessed" in order to obtain the requisite specific heats and equilibrium constants. This process is repeated until the "guessed" and calculated temperatures agree to the required accuracy. From this result the specific internal energy of the mixture per unit weight is arrived at. The gain in specific internal energy is a measure of the work done on the working substance during compression. The next steps involve the determination of the internal energy of the hot products of combustion at the beginning and end of the expansion stroke. The total loss in internal energy represents the work done per cycle and this, divided by the heat input, gives the theoretical thermal efficiency. The nett heat value of the fuel is taken, since the gross figure gives an unattainable ideal.

For the ideal Diesel engine, the solution is arrived at in a similar manner, with certain modifications. External work is done by the mixture during combustion, giving a lesser gain in internal energy than in the constant-volume cycle. The volume at cut-off must be determined before the final temperature and internal energy can be estimated.

The results are expressed in the form of tables and curves for three fuels for the Otto cycle (gasoline, "90 per cent." benzole and blast-furnace gas) and for "petroleum oil" for the Diesel cycle. The range of compression ratios covered is sufficient for all practical purposes. For each compression a range of air-fuel mixtures is dealt with, from considerably excess air down to the theoretical amount for complete combustion. The ideal efficiencies of the different fuels on the Otto cycle are almost identical, the blast-furnace gas giving slightly the lowest figures. In all cases the efficiency is increased by a

high compression ratio and a high percentage of air. For this reason, the calculated thermal efficiencies of the Diesel cycle are higher than those for the Otto, because of the higher compression ratios and air-fuel ratios considered in the former calculations.

Curves are given showing the large discrepancy between ideal efficiencies calculated in the above manner and those based on a "cold-air" standard which assumes the working substance to be a gas whose thermal properties approximate to those of air at normal atmospheric temperature. Empirical "hot-air" standards are deduced to fit the curves obtained for the various fuels. A method of evaluating the mean effective pressures of the different cycles is outlined.

Engine efficiencies can be expressed in several ways which give the same numerical result. Thus the engine efficiency

$$= \frac{\text{Brake thermal efficiency of actual engine based on L.H.V.}}{\text{Ideal cycle efficiency based on L.H.V.}}$$

$$= \frac{\text{Brake thermal efficiency of actual engine based on H.H.V.}}{\text{Ideal cycle efficiency based on H.H.V.}}$$

$$= \frac{\text{fuel required for ideal engine, lb. per H.P./hour.}}{\text{fuel used by actual engine, lb. per B.H.P./hour.}}$$

$$= \frac{\text{brake M.E.P. of actual engine}}{\text{M.E.P. of ideal cycle}}$$

where L.H.V. and H.H.V. represent nett and gross heat values respectively.

Four sets of test results are given for different Otto engines, one set for a marine Diesel engine. The engine efficiencies at different air-fuel ratios are plotted graphically along with observations of B.H.P., mechanical efficiencies and specific fuel consumptions. The maximum engine efficiencies are obtained with mixtures weaker in fuel than those for maximum power and vary from 50–60 per cent. for the small-and moderate-powered Otto units to 76 per cent. in the case of a 185 H.P. aero-engine. The Diesel gave a low figure, 61 per cent., owing to its low mechanical efficiency.

The conclusion is reached that, in spite of the difficulty and the problematical assumptions involved, the calculations show very clearly the necessity for some other standard than that of "ideal-air" on which to base engine performance. The suggestion is made that some of the results, or a suitable combination with those of Goodenough and Baker (*Bull.* No. 160, University of Illinois, 1927) and of Tizard and Pye (*Automobile Engineer*, Feb., 1921, p. 58), may be useful as standards. The advantages of quoting engine efficiencies rather than indicated or brake thermal efficiencies are outlined, particularly for sales purposes. G. B. M.

646. The Possibility of Increasing Fuel-Economy. H. M. Jacklin. *S.A.E. Journal*, 1929, **25**, 159–163.

The author compares the efficiency of a 3 in. by 4 in. petrol engine operating under various load conditions at constant compression ratio with fixed ignition, constant compression ratio with variable ignition, and constant compression pressure with variable ignition. He shows that the performance improves as conditions are altered in the above order. When the results are applied to a car the curves show that the advancing of the spark at low loads results in a gain of 1½ to 2 miles per gallon and that working at constant compression pressure improves the consumption by about 8 miles per gallon.

The car selected for the comparison required 15 H.P. at 45 m.p.h. and 62 H.P. at 70 m.p.h. for propulsion on level concrete road without wind. R. S.

647. EXPERIMENTAL COMBUSTION CHAMBERS FOR HIGH SPEED DIESEL ENGINES. C. Kemper. *Oil and Gas Power*, 1928, **50**.

The design of combusion chambers to meet the needs of high speed Diesel engines for aeronautical work is considered, and it is postulated that the most important requirements are that the fuel spray shall be completely distributed through the chamber and that the degree of turbulence shall be sufficient to produce complete combustion in the time available. Tests of various shapes of combustion head have been made on a single cylinder engine of 5 in. bore and 7 in. stroke. The fuel injection system consisted of a primary gear pump supplying oil at 90 lb. per sq. in. to a cam actuated impact type of injection pump and an automatic injection valve, the static opening pressure being 6,000 lb. per sq. in. The fuel used was a Diesel grade having a specific gravity of ·847 at 80° Fah.

Maximum pressures were determined by means of a diaphragm type of valve operated by cylinder pressure, the pressure of the gases trapped above the diaphragm being measured by a pressure gauge. The preliminary tests were made with a compression ratio of 11·4 to 1, in a standard Liberty aircraft engine cylinder, the design being such that it was difficult to arrange an injection valve which would permit of fuel reaching all the air in the combustion chamber. When working at full load it was necessary to inject 50° before top centre to obtain reasonably good combustion and this led to explosion pressures of 1,300 to 1,600 lb. per sq. in. gauge. Aluminium pistons were used and had a very short life, the high pressures cracking either the crown or the gudgeon pin bosses. Curves of power output and consumption are given, the power being 34 per cent. less at 1,750 R.P.M. than that obtained from the "Liberty 12" standard cylinder operating on petrol at the same speed. The fuel consumption, however, shows an improvement of about 6 per cent. The second series of tests was made with a special head of flat design necessitating the use of a fan-shaped spray of fuel. With this it was possible to work with a maximum pressure of 800 lb. with smoother running and with freedom from piston trouble, but the performance was poor. Experiments to improve combustion by directing the flow of air through the inlet valve enabled the mean pressure to be increased from 82 to 96 lb., and with decrease in consumption from ·60 to ·51 lb. per I.H.P. Hr. The flow was so arranged as to direct the air towards the fuel valve, thereby removing products of combustion left from the previous cycle. The third head used was arranged with a bulb type pre-combustion chamber and had a compression ratio of 9·9 to 1. This made starting from cold difficult without jacket or inlet air heating. The fuel system consisted of an eccentric operated pump and a diaphragm type injection valve, and the design gave a B.M.E.P. at full load of 71 lb. per sq. in. (I.M.E.P. 106 lb.). The consumption was ·42 lb. per I.H.P.Hr., when the excess air was 15 per cent. At higher fuel air ratios, the curves flatten indicating imperfect burning. The fourth head examined was one with a pear-shaped bulb designed to give a high degree of turbulence in the bulb during compression and in the cylinder during expansion. This was obtained by arranging a 9/16 in. orifice tangentially to the bulb to give directional control. When the piston was on top centre, the ratio of air volume in the bulb to that in the cylinder was approximately 1 to 1. The compression ratio was 13·5 to 1, giving a 450 lb. gauge pressure. The head operated smoothly under all loads, and at 1800 r.p.m. the B.M.E.P. was 85 lb. per sq. in. with a mechanical efficiency of 80 per cent. The fuel consumption was ·60 lb. per B.H.P. Hr.

It is concluded that the speed of airless injection engines may be increased by controlled turbulence without encountering excessive explosion pressures. It is not, however, possible to increase the mean effective pressure to that of carburetter fed petrol engines without a reduction of the time lag of combustion, and this is believed to depend on the development of better means of preparing the charge before it enters the cylinder. R. S.

648. Hydro-Pneumatic Reservoir for Petrol Fuels and the like. M. Baccich and C. D. Filippi. E.P. 290,663. May 7, 1928.

A hydro-pneumatic tank for the supply of fuel under pressure is constructed so that the fuel and water tanks are combined in one integral structure and connected by an air chamber. The air chamber acts as a pneumatic cushion for maintaining a constant pressure on the fuel and an automatic cut-off prevents the entry of water into the fuel chamber. The cut-off is actuated by a float in the water chamber. G. S.

649. Petrol Tanks for Aeroplanes, Motor Cars and such like. W. T. Caddy and D. Doig. E.P. 316,117. April 23, 1928.

A flexible liquid-tight tank has its walls composed of inner and outer laminæ of fabric, between which ribbons or bands of flattened wire coils, served with rubber to fill all the intestices, laid side by side and pressed to form sheets. The whole structure is shaped to form a tank and vulcanised. The rubber served or filled bands may be enclosed in rubber sheets and made one with pressure. G. S.

650. Removing Oil, Grease and the like Foreign Matter from the Interior Surfaces of Steam Condensers. S. B. Freeman and Imperial Chemical Industries, Ltd. E.P. 315,930. May 4, 1928.

The degreasing of the interior of steam condensers or other structures not easily accessible is carried out by applying the vapour of a volatile solvent, particularly chlorinated hydrocarbons such as trichlor ethylene, condensing it *in situ* and removing the solution. The vapour may be produced by evaporating the solvent in the vessel to be cleaned or in an external vessel, and the condensation may be effected by a variety of methods. G. S.

651. An Improved Process for Producing Non-Corrosive and Heat Resisting Surfaces on Iron. J. Y. Johnson, Assr. to I. G. Farbenind. Akt. Ges. E.P. 314,314. June 19, 1929. Appl., March 19, 1928.

The use of aluminium cementation in protecting the surface of iron against corrosive agents and destruction by heat is already known. It has already been proposed to alloy the surface of iron articles by heating them in an inert or reducing atmosphere with a mixture of finely powdered chromium and aluminium alloy in the presence of alumina or chromic oxide.

This invention claims to speed up the alloying process and to produce a better surface by treating the articles at a temperature above 800° C. in an inert or preferably reducing (hydrogen) atmosphere in contact with an aluminium-chromium alloy not mixed with other solid chemically inert material.

It is claimed that the resulting superficial alloy on iron is inseparably combined with the iron giving a surface free from pores which can be polished and is easily worked with a file or in a lathe. Any thickness of diffusion layer can be obtained at will by modifying the temperature and duration of the heating. G. B. M.

652. HYDRAULIC SYSTEM FOR STORING AND DELIVERING OIL. W. J. Peter E.P. 316,109. July 22, 1929. Appl., Jan. 21, 1928.

Such a system, in which the storage tank is always filled with liquid: oil and/or water, is not new. The present invention relates to improvements in such systems, especially those adapted to marine use, whether for handling oil in bulk on a tanker, or for handling bunker oil for the boilers or engines. The advantages claimed for the invention include the following:—

1. Positive control of all oil; ready transfer of oil and water to and from all parts of the system, or overboard.

2. Automatic shutting of the inlet valve when water falls to a predetermined low level during fueling.

3. Automatic shutting of the discharge valve when water rises to a predetermined high level during delivery.

4. Automatic care taken of expansion and contraction, the tanks always being maintained full of liquid.

5. Absence of any vapour space in tanks, hence absence of explosion risks, reduction in deposits on walls, and increased stability during rolling and pitching.

6. Easy transfer to another tank of the oil normally remaining in the head when necessary for tank cleaning or refilling with a different oil.

7. Water clearing of the tank by water pump prior to cleaning.

Each tank is provided with a "service head," from which depends an upper ball float valve designed to sink in oil but float in water (in the latter case closing the oil delivery valve), and a water leg reaching to the bottom of the tank where it supports a similar float, which closes the oil inlet when it sinks in oil. Each service head is connected to a centrally located oil control manifold, having two valves per tank: one connecting with the common fueling line, the other connecting with the common discharge line to the service tank and booster pump. Each water leg is connected to the central compartment of a two-way water control valve consisting of three compartments, the forward compartment being supplied with sea water from the pump under a pressure limited by an elevated overflow pipe; the rear compartment connecting to (*a*) a bleeder or make up line supplied from the pump *via* a sight feed, and (*b*) the overflow line mentioned above. The central compartment can be opened up to either one or other of the end compartments by means of a single lever operating a double valve. The water leg is put in communication with the forward compartment only when forcing water into the tank. When refueling or standing idle the water leg is opened up to the rear compartment, excess of water passing to the overflow, together with the make-up supply, which latter can flow back to the tank to compensate for any contraction. These water control valves form a centrally located battery.

The shallow bottom tanks are provided with their service head at the top of an "instead" tube, level with the service heads of the wing tanks. This eliminates intermittent delivery during rolling and pitching. The small amount of oil remaining in the service heads after delivery can, when necessary, be transferred to another tank *via* the vent pipe of each tank.

By means of a two-way valve similar to those of the water control manifold, the suction of the pump can be changed over, prior to cleaning a tank, to the bleeder line so that by proper manipulation of the valves, the water from any desired tank can be withdrawn *via* the rear compartment of the water control valve, and discharged overboard or to any other tank.

Nine diagrams and a full description of the apparatus and its working are given. L. H. A.

653. CLEANING THE TUBES FOR THE PASSAGE OF HEATED GASES OR PRODUCTS OF COMBUSTION IN STEAM AND OTHER BOILERS AND THE LIKE HEATED BY WASTE HEAT OR OTHER HEATED GASES OR PRODUCTS OF COMBUSTION MADE TO PASS THROUGH TUBES THEREIN. J. Summers and Sons, Ltd., and F. A. Hibbs. E.P. 315,732. April 16, 1928.

A device for the cleaning of boiler tubes comprises guide tubes mounted on a supporting part, *e.g.*, a plate or smoke-box door, in a position opposite to the ends of the tubes and of sufficient length to provide adequate guidance. The cleaning rods, etc., are provided at suitable distances with parts so to fit the guide tubes that they are guided to enter the tubes to be cleaned with certainty. Suitable means are provided for closing the guide tubes when not in use as such. G. S.

See also Abstracts Nos. 598, 656 and 657.

Anti-Detonation.

654. THE MEASUREMENT OF DETONATION IN INTERNAL-COMBUSTION ENGINES. R. O. King and H. Moss. *Engineering*, **128**, 1929, 219–221 and 272–274.

In a preliminary section the authors show that the cost of adding anti-knock material to a low value spirit to enable it to be used in an engine of higher compression ratio may be such as to offset the saving due to increased efficiency. In the case of benzole there is a slight but progressive improvement, but with ethyl fluid an economic maximum is reached in the neighbourhood of 6 c.c. per gallon for the example selected. This is due to the fact that the improvement possible with the metallic dope tends to reach a limiting value. Reference is made to the inter-relation between tendency towards detonation and the content of hydrocarbons of the paraffin series present, and also to the connection between detonating tendency and the formation of peroxides, but it is concluded that direct measurement in an engine is the only method available at present. Three general classes are dealt with. Method 1 depends on the use of a bouncing-pin indicator, and fuels are compared by measuring the amount of anti-knock material which must be added to each to bring it up to a given standard. Method 2 is similar, but an audibility method of detection of the detonating value is used. Method 3 consists of the determination of the H.U.C.R. Methods 1 and 3 are compared in detail, and the operating conditions under which the tests were made are fully described. Experiments made with a bouncing-pin indicator in the Ricardo E 35 engine showed that the instrument was less sensitive than the ear in detecting the beginning of detonation, and that in this engine it was not suitable for use over a range of compression ratios.

An examination of the consistency and sensitivity of the bouncing-pin method used in a Delco engine showed that certain fuels gave more irregular results than others, and that the experimental error of the particular routine adopted was about equivalent to 2 per cent. benzole. The H.U.C.R. method gave results in agreement to about ·05 ratio, provided tests were always compared against a stock reference fuel. The error tended to become greater at ratios higher than about 6·0 to 1.

Curves are given showing the effect of additions of ethyl fluid to various blends of Californian spirit and naphtha when tested by the two methods, and the comparison of results shows that the difference may reach 3·4 c.c. of ethyl fluid with high concentrations of the metallic dope. The difference between values over the range of concentration used in practice reaches 1 c.c. per gallon. When the differences are expressed in terms of compression ratio only one pair of fuels show a figure greater than 0·1 ratio. R. S.

655. Flame Characteristics of "Pinking" and "Non-Pinking" Fuels. Part II. G. B. Maxwell and R. V. Wheeler. *J. Inst. Petr. Techn.*, 1929, **15**, 408–415.

Observations on the explosions of pinking and non-pinking fuels in a closed cylinder reported in Part I (*J.*, 1928, **14**, 175) have been confirmed and extended by experiments with other fuels and fuel-blends. The requisite fuel vapour-air mixtures, under given initial temperatures and pressures were exploded in a closed horizontal cylinder fitted with a glass window along one side and an optical pressure indicator at one end. A continuous record of the flame movement and pressure fluctuations throughout the explosion was obtained on a strip of sensitive bromide paper wound round the periphery of a drum which was rotated at constant speed. A number of records (negatives) are reproduced in the paper.

The experiments on *iso*-pentane and benzene, reported in Part I, were repeated at higher initial temperatures (up to 100° C.). The effect of increased temperature on *iso*-pentane was small. A slightly wider range of mixtures gave an audible explosion and showed the sudden pressure increment and shock-wave remarked before, but the effects were not more violent than at 15° C. Benzene-air mixtures at 30° C. and above differed from the same at 15° C. At the higher temperatures, and pressures of 2 atms. and above, certain rich benzene-air mixtures gave an audible explosion, but no maintained shock-wave. As in the earlier work, the combustion of benzene-air mixtures was observed to be continuous and long continued behind the initial flame. Records obtained with pentane-benzene blends clearly show the difference in the combustion of the two fuels. "Auto-ignition," that is the ignition of unburnt charge by heating and compression due to the combination of the remainder of the charge, was observed in certain blends. Such auto-ignition had no magnifying effect on the subsequent pink and can hardly be the cause of pinking in an engine cylinder.

The effect of two "pro-knocks" (ethyl ether and amyl nitrite) is shown to be similar to that in an engine cylinder. Incombustible gases such as carbon dioxide and steam have an "anti-knock" effect, that of steam being greater than can be accounted for by its cooling influence.

Explosions of a variety of other fuels, the behaviour of which in an engine is known, support the view that, for a pink to occur, sufficient residual energy must be latent in the products of the initial flame to maintain a subsequent shock-wave.

The circumstances leading up to the formation of a shock-wave are made somewhat clearer by the observation of a stationary pressure wave, *i.e.*, of resonance, in the hot gases towards the end of flame travel whenever a subsequent pink occurred. Resonance led to acceleration of flame speed and rate of combustion followed by a recoil when the flame reached the solid end of the cylinder. Such a recoil, travelling back as a pressure wave and being maintained by a release of energy from the partially burnt products would be similar to a "retonation wave" and could easily give rise to the sound of a pinking explosion when it struck the walls of the combustion space. G. B. M.

656. The Influence of Cylinder Design on Pinking. G. B. Maxwell and R. V. Wheeler. *J. Inst. Petr. Techn.*, 1929, 15, 415–427.

From observations described in previous papers ("Flame Characteristics of Pinking and Non-Pinking Fuels," Parts I and II) there appear to be two principal ways of eliminating "pink." (1) As regards the fuel, once ignited its continuous combustion could be encouraged (*e.g.*, by blending or doping) so that little residual energy is available to maintain a shock-

wave. (2) As regards the engine, the shape and size of combustion-space, the position of the sparking-plug, the arrangements for causing turbulence could be so designed as to prevent the initiation of resonance in the gas charge. The present paper describes closed-vessel experiments on such engine factors. The results show that the factors which militate against the setting-up of resonance are those which tend to eliminate pinking in an engine cylinder. Turbulence inhibited the formation of a stationary wave; a shorter length of flame travel, a greater cooling surface exposed to the flame, a spherical combustion space, all had the same effect. In addition, the contour of the end-plate against which the initial flame impinged had a slight effect. It was also observed that restrictions in the combustion space placed near the point in the flame travel where resonance invariably first made itself felt had a distinct anti-pinking influence on the subsequent course of the reaction. G. B. M.

657. CYLINDER HEAD DESIGN. H. R. Ricardo. *Automobile Engineer*, 1929, **19**, 257–261 and 284–286.

The objects aimed at in the original design of Ricardo "turbulent head" design for side valve engines are summarised as follows:—

1. Creation of additional turbulence during compression to:—
   (*a*) Increase rapidity of burning, thus obtaining a greater effective expansion ratio and reducing sensitivity to ignition timing.
   (*b*) Reduce tendency to detonation by keeping unburnt portion of charge in rapid motion by breaking up flame front.
2. Reduction of maximum flame travel by suitable placing of plug.
3. Reduction of length of flame travel from plug to farther end of combustion space by arranging that the piston closely approaches the head over a large part of its area.
4. Reduction to a minimum of the surface volume ratio.

Combustion in an engine cylinder starting from a line of flame across the plug electrodes spreads to the immediately surrounding mixture at a rate depending mainly on the temperature of the flame front, and to some extent on the density of the charge. The combustion, at first gradual, rapidly increases in speed and the turbulence breaks up the resulting self-propagating nucleus of flame, dispersing it through the compression space and enormously accelerating the combustion.

As regards detonation, it seems clear that this starts only at the extreme end of flame travel and is due to the compression of a small portion of unburnt charge by the approaching flame. With a given fuel, the absence or occurrence of detonation depends on whether this unburnt gas can get rid of its heat to the walls at a sufficient rate. This depends on the length of flame travel (which determines the speed of the oncoming flame), on the temperature of the walls surrounding the unburnt residuum, on the degree of turbulence, and on the effect of hot spots in the combustion chamber which may raise the temperature of the cycle by inducing local surface combustion during the early parts of the cycle. Experiments have been carried out: (*a*) on a special single stroke compression engine with an adjustable degree of turbulence and with firing either by plug or by compression ignition; (*b*) on a special sleeve valve engine arranged with quartz windows in the head so that flame spread can be observed through a stroboscope; (*c*) on a variable compression engine in which the effects of various combinations of valves and plugs can be tested; (*d*) on a variable compression engine with turbulence controllable by means of inlet port vanes; and (*e*) on a number of other research engines of various types. The more recent experiments have shown that the rate of burning depends on the degree of turbulence and may be expressed in terms of increase of pressure

per degree of crank angle. The rate depends to a lesser degree on the compression ratio. At any compression ratio the best output and efficiency are obtained on full load when the pressure rise is about 30 lb. per sq. in. per degree of crank angle. Below 25 lb. per sq. in. the loss by delayed combustion is sufficiently serious to cause heavy carbonisation and burning or pitting of valves with sensitivity to ignition timing and tendency to fire back into the carburetter if the mixture is weakened. Above 35 lb. per sq. in. performance begins to fall owing to convection losses increasing, and, in addition, the engine running becomes rough and noisy.

In a turbulent head the turbulence is produced almost equally by the initial velocity through the inlet valve and the velocity through the restricted throat into the compression space during compression, and it is intensified by the rapid movement of the mixture trapped above the piston at the end of compression. Turbulence from each source increases with engine speed, so that the ratio of rate of pressure rise to degrees of crank angle remains almost constant at any speed.

In all cases, the best possible location of the plug is nearly in the centre of the effective combustion space but slightly set towards the exhaust valve. The effect of clearance between the piston head and the cylinder is critical as regards tendency to detonate, a reduction from 0·15 in. to 0·10 in. enabling the compression ratio to be raised by 0·85 ratio in the example tested.

The effect of turbulence alone on detonation is appreciable, but it is not so marked as the effects of length of flame travel and throat area. The effect of alteration of inlet valve area is similar to that of alteration of throat area in the head. Stream-lining of the head from the throat was *not* found to give any useful advantage as compared with a rectangular section. Uni-directional turbulence gives very poor results compared with the indiscriminate type.

It has been found that the ordinary turbulent head has certain limitations, the chief being that the high degree of turbulence leads to abrupt application of pressure with the consequent spring of moving parts and high period vibrations known as "roughness." This can only be avoided by the use of an exceptionally rigid design of engine, and a two-bearing crankshaft is unsuitable for even 20 lb. per sq. in. rate of pressure rise. Recent research has shown that roughness is due, not to the actual rate of pressure rise, but to the abrupt application of the pressure, and if the pressure is first applied gradually, a very rapid rate may be used later without trouble. It has been found that if about 15 per cent. of the total charge be isolated and kept stagnant, and thus burnt slowly after the passage of the spark, the remaining 85 per cent. can be burnt with a 50 lb. per sq. in. rate without serious roughness. This effect is obtained by forming the head with a shallow flat-roofed pocket, usually over the inlet or exhaust valve, and placing the plug above this pocket. This design is known as the "shock-absorber" type. The ignition advance for this type is insensitive and much greater than for the ordinary turbulent design. Results are very slightly better than for the ordinary type apart from the greatly increased smoothness of running. A second design known as the "high power" head is intended for high speed engines. The combustion space is offset and is of the domed type, the exhaust valve fills the whole of the space usually occupied by both exhaust and inlet valves, the inlet valve opens downwards into the cylinder in the flat portion between the piston and head, and any degree of shock absorber action may be employed. Amongst other advantages, the resulting design gives high volumetric efficiency with short flame travel, and the turbulence is independent of the size of the inlet valve.

The author warns designers against the unconsidered use of high compression ratios made possible by high turbulence, and emphasises the fact that comparatively small gain in performance is accompanied by very large increases in stresses which may result in destruction of working parts unless these can be specially designed for the heavier duty required of them.

The articles are illustrated by numerous diagrams embodying the experimental data on which the conclusions arrived at are based. R. S.

658. Effects of Knock-Suppressing and Knock-Inducing Substances on the Ignition and Partial Combustion of Certain Fuels. R. E. Schaad and C. E. Boord. *Ind. Eng. Chem.*, 1929, **21**, 756.

The minimum igniting currents for hot-wire ignition of toluene, isoamyl acetate and kerosine have been determined for a range of fuel-air strengths, the igniting source being an electrically heated platinum or palladium filament. Fuel-air ratios were varied by varying the temperature of a given volume of fuel in a saturator through which pure, dry air was passed at a fixed rate. Under these conditions it was found that the most easily ignitible mixture of each fuel contained only sufficient oxygen for combustion of the carbon to carbon monoxide. The addition of knock-suppressors raised, and of knock-inducers lowered, the minimum igniting current for each mixture.

The hot-wire method was supplemented by the method of break-spark ignition, effected by breaking a D.C. circuit between brass contacts. In this case the most readily ignitible mixture for each fuel was that containing sufficient oxygen for complete combustion to carbon dioxide. The addition of dopes had little if any effect. The contrasted results obtained by the two methods led to the conclusion that there was "pre-ignition combustion" on the surface of the wire in the first set of experiments and that this was the phase affected by the dopes. Proof of pre-ignition combustion was obtained by analysis of the mixtures passed over the hot filament when the current through the latter was insufficient to cause ignition. No carbon monoxide was detected but carbon dioxide was formed in all cases. Without exception the addition of knock-suppressors decreased and of knock-inducers increased the pre-ignition combustion as measured by the carbon dioxide content. Repeat experiments, with the same filament, on the undoped fuels after a series on the same fuels doped, checked sufficiently closely to prove that the effects were not due to surface poisoning or activation of the hot wire. The dopes had the most marked effect, for a given fuel, on that mixture which was most easily ignited by this method.

A short discussion is appended on similar work by other investigators on the effect of dopes on igniting temperatures and rates of oxidation.

G. B. M.

659. Performance of Gasoline in Engines (Spectrum Analysis of Combustion in Engine Cylinders). W. C. Thee, *Oil and Gas J.*, **28**, 12, 1929, 46.

A short description is given in an introduction and appendix to this paper of the origin and variety of spectra, their classification and measurement and of the method and application of spectrographic analysis.

A typical series of emission spectrographs is shown representing detonating and non-detonating combustion in an engine cylinder of a straight-run gasoline and one of the non-detonating combustion of benzene Detonation gives an intense continuous spectrum upon which is superimposed an ill-defined band spectrum. Benzene under non-detonating conditions gives the same results but gasoline under these conditions shows no continuous spectrum. The author concludes that, from our knowledge of the combustion of carbon

monoxide with and without steam or hydrogen, the banded and continuous spectra of a detonating combustion are associated with the combustion of carbon monoxide in the cylinder. G. B. M.

660. Improvements in and Relating to Fuels for Internal Combustion Engines. British Dyestuffs Corporation, Ltd. E.P. 287,192. March 22, 1928. Appl. Oct. 25, 1926.

The invention relates to a method of suppressing "knocking" or "pinking" in internal combustion engines. A heavy metal derivative of a β-diketone of the type R.CO.C—R_1, .COR_2, in which R_1 and R_2 are hydrocarbon radicals, is added to the motor fuel. The β-diketone may conveniently be, acetylacetone, propionylacetone or β-methyl-acetylacetone, while the metal present may be chromium, iron, cobalt, manganese, or therium. H. E. T.

661. Improvements in or Relating to Processes of Producing Tetraethyl Lead. W. S. Calcott, A. E. Parmalee, and F. R. Lorriman. E. P. 280,169. Oct. 18, 1928. Appl. July 26, 1927.

A process is described for producing lead tetraethyl by effecting a reaction between an alloy of lead, sodium and potassium and ethyl chloride in a closed reaction vessel at a temperature of 30° to 45° C. H. E. T.

662. Improvements in or Relating to Fuel for use in Internal Combustion Engines. A. C. Egerton and C. H. Barton, Assr. to Asiatic Petroleum Co. E.P. 300,156. Nov. 6, 1928. Appl. Aug. 6, 1927.

This invention relates to improvements in fuel for internal combustion engines with the object of preventing or minimising pinking. The distinctive feature is the use of a petrol-soluble compound of an alkali metal, particularly potassium oleate or naphthenate, in amounts of from 0.5 to 1.5 per cent. of the fuel by weight. This invention is distinct from the use of soap emulsifiers for heavy oils, or the use of petrol-soluble salts of organic acids to prevent iron oxide deposit in fuels doped with iron carbonyl. The invention is restricted to the use of the above mentioned compounds to dope hydrocarbon fuels, such as petrol, for the specific purpose of overcoming or minimising pinking. G. B. M.

Cracking.

663. Some Aspects of Cracking. A. E. Dunstan and R. Pitkethly. *Ind. Eng. Chem.* 1929, **21**, 643.

Liquid phase or pressure distillation processes have been responsible for the enormous production of cracked gasoline in recent years, and it is remarkable that the few processes which have become commercially successful have all developed on similar lines—namely, the provision of a tubular heating section and a large vessel wherein the reaction is completed and coke deposited. All rely on heat treatment for the decomposition of the larger molecules, and they can process almost any crude or residual oil for the production of motor spirit. It would appear that motor fuel requirements can be satisfied for an indefinite time simply by increasing cracking equipment. The ideal process, however, has not yet been invented, for by heat treatment alone heavy hydrocarbons may be cracked to light hydrocarbons, but it is impossible to do this without also producing coke or compounds of high carbon and low hydrogen content. The amount of coke produced in liquid phase cracking may be only a small percentage of the raw oil processed, but last year over a million tons were produced in the United States alone. The coke problem

demands attention, and one method of reducing this is by imparting a high degree of turbulence in the oil stream without decreasing the time factor, thus reducing the difference in temperature between the oil and the walls of the containing tubes.

From the same raw oil the products obtained by cracking in the liquid phase and with vapour phase differ greatly in composition, liquid phase gasoline being similar to the straight run product whilst the vapour phase spirit has very high antiknock value.

By developing both processes it appears possible to supply quantity and quality requirements by a balanced production of liquid and vapour phase spirit from the same raw material in order to supply the type of fuel necessary for the best results in the modern high-compression, high-speed engine. It is reasonable to expect that the cracking plant of the future will comprise both liquid and vapour phase steps. Generally the same types of compounds are present in both liquid and vapour phase products but in different proportions.

Much work has been done during the last few years in the study of the physical and engineering side of cracking, resulting in efficient modern plant, but there is a lack of knowledge of the chemical changes involved in the cracking process and of the chemical nature of the resulting products, and more research work on these subjects is universally demanded. R. P.

664. VALUATION AND CRACKING OF GAS OILS. R. H. Griffith. *J. S. C. I.*, 1929, **48**, 252–263T.

This investigation was carried out in order to find a method of analysis which would classify oils with regard to their value in gas making, as no satisfactory work has been done in the cracking of a wide range of oils. The results obtained were arrived at primarily, by measuring the number of British thermal units obtained in the form of permanent gas from a known amount of oil. This was done by direct determination of the calorific value of the gas, produced by cracking in a stream of hydrogen or other gas.

The paper is divided into six sections:—(1) description of method and apparatus; (2) results obtained with varying conditions and the same oil; (3) results obtained by cracking different oils under comparable conditions; (4) method of analysis of oils and correlation of chemical composition with behaviour on cracking; (5) tar formation; and (6) results on water gas plant.

The apparatus consisted of a silica tube wound with nichrome wire and well insulated against heat loss. Rheostats and the necessary instruments controlled the temperature. The upper part of the tube was filled with steel balls supported on a grid to ensure proper vaporization of the oil. A thermocouple passed through the grid and extended to the centre of the cracking tube. A hydrogen cylinder was connected, through a flow gauge, to the top of the cracking tube, and a water-cooled condenser and tar separator connected to the bottom of the tube. The bulk of the tar collected in this separator and the gas was freed from tar fog by passing through glass tubes containing wood shavings and cotton wool. A meter and Boys calorimeter completed the apparatus.

The effect of changes in the rate of feed and of alterations in the surface were studied. The effects of temperature on one particular oil were determined by running at 650°, 700°, 750°, 800° and 850°. In each case the maximum yield in therms per gallon was determined and the yield of tar at each temperature recorded. The effects of adding hydrogen and nitrogen in plain and packed tubes were investigated, tar formation received attention and comparative tests made with different oils. Finally, tests on a water gas plant were carried out. The method of analysis developed is described in a paper already published (*J. S. C. I.*, 1928, **47**, 21T).

The yield of gaseous therms per gallon rises to a maximum and then decreases steadily with increase in the rate of feed.

Temperature changes cause considerable differences in the efficiency of the plant. In every case the best temperature was found to be 750° to 760°. There is no doubt that in water gas practice the most important factors are the rate of oil feed and the temperature of the carburetter and superheater. Variations in these two factors are more noticeable than alterations in the composition of the oil. The quantity and quality of the tar formed vary widely with variation in the cracking conditions. The yield of the tar falls to a minimum then remains practically unchanged with increase in the rate of feed. The paraffin content is high at low rates of feed and the naphthalene content reaches a maximum at the same time as the yield of therms per gallon is highest. R. P.

665. The Vaporisation of Petroleums and Pressure Distillates. E. H. Leslie and G. H. Faucher. Presented before the Petroleum Division Amer. Chem. Soc., Sept., 1928.

This paper contains results obtained in an investigation on "The Determination of the Physical Properties of Petroleum and Petroleum Products." Two crude petroleums were used in this investigation, one representative of the mixed-base Mid-Continental crude oils and the other typical of the cyclic-base Californian crude oils of low gasoline content. Two pressure distillates were also studied, one representing the distillate from shell-type pressure stills, and the other the product from a pipe still cracking unit using heavy fuel oil as charging stock. These four materials were flash-vaporised under equilibrium conditions and the properties of the resulting distillates and residuums were determined.

The material to be vaporised flows from a reservoir to a constant head device and from thence through a heating coil to the equilibrium chamber. This chamber is maintained at any desired temperature by means of a thermostat. The materials were flash-vaporised in substantially the same way as that employed by Leslie and Good (*Ind. and Eng. Chem.*, 1927, **19**, 453). From the results obtained during this research the following conclusions may be drawn.

1. The amount of single-flash distillate boiling below the flash temperature averages 80·8 per cent. for a typical Mid-Continental petroleum.

2. The amount of single-flash residuum boiling over flash temperature averages 8·9 per cent. for a typical Mid-Continental petroleum and 7·9 per cent. for a heavy Californian petroleum.

3. Materials such as pressure distillates, which contain a limited range of components, behave too anomalously to predict exactly the characteristics and behaviour of their flash-vaporisation products.

4. No simple relation was found between the distillation curves of the single flash products from petroleums or pressure distillates. H. E. T.

666. Manufacture of Commercial Anhydrous Aluminium Chloride. A. M. McAfee. *Refiner*, 1923, **8**, (8), 57–60.

The chief difficulty lies in overcoming the erosion of retort walls by chlorine at 1600° F. The following process has been operated by the Gulf Refining Company for several years.

Crude bauxite ore is calcined in an internally heated rotary kiln at about 1800° F. to drive off moisture. It is then mixed with coking coal in the proportions of one part coal to three of bauxite by weight. The mixture is pulverized, mixed with a liquid binder, briquetted, and the briquettes pre-

heated to about 1500° F. to expel all volatile matter, leaving a hard briquette of approximately 82 per cent. bauxite and 18 per cent. carbon. These hot briquettes are charged to the vertical chlorinating furnace, which is built up of two courses of firebrick encased by an iron jacket, the latter being protected by an internal lining of powdered bauxite. The charge is raised to chlorinating temperature (about 1600° F.) by a short blow of air, after which chlorine is passed downwards through the charge for 8–10 hours. Aluminium chloride passes from the bottom to a firebrick cooler from whence it is discharged into the condenser proper, consisting of vertical 16-inch iron pipes provided with scrapers. The result is that the chemical is deposited in hoppers as a powder, 75 per cent. passing a 10-mesh screen. The chlorinating furnace lining is good for 100 days and the condensers have a life of two years, attributed to a protective coating of aluminium chloride.

Recent improvements eliminate briquetting, substitute refinery coke for coal, eliminate air blasting of the chlorinating furnace, and permit larger furnaces having a longer life.

Impurities in the crude bauxite ore must be watched as they are all converted into volatile chlorides. The dried bauxite should contain not more than 5 per cent. of silica nor more than 3 per cent. of iron oxide. At present aluminium chloride of 94 per cent. purity can be produced and marketed at 5 cents. per pound in car-load lots. L. H. A.

667. Chemism of Cracking. Ssachanen and Tilitschejev. Ber., 1929, 658–677.

The experimental work on which this study is based began with the cracking of paraffin (m.p. 53°) in an autoclave. The liquid products were fractionated, the unsaturated bodies removed from each and the remaining material found to consist almost entirely of paraffins. A sample of di-isoamyl was cracked at 425° at a pressure of 83 atmos. for 63 mins. The gas contained 20 per cent. of hydrogen, suggesting that with the iso structure and tertiary carbon atom, the hydrogen splits off more easily than from the normal paraffins. That the chain tended to break near the middle was indicated by the high proportion of pentane and hexane found in the fractions. The lower olefines were probably polymerised to some extent, but were present only to the extent of 22.9 per cent. instead of the 50 per cent. to be expected. The aromatics chosen for cracking were naphthalene, anthracene and cymol. The first, even when heated to 475° at 25–50 atm. for 4 hrs. 20 mins., only formed higher boiling condensation products, which were somewhat darker in colour and contained no coke. Anthracene was much less stable than naphthalene, for on cracking at 70 atm. for 2 hrs. 37 mins., 59 per cent. of the product was coke. The presence of anthracene and other highly condensed hydrocarbons in the heavier fractions of petroleum is taken to be the explanation of their tendency to form coke when cracked. Cymol was heated at 425° and 85 atm. for 3 hrs., but only about 10 per cent. of the original substance was decomposed, the velocity of cracking being only 1/10 that of di-isoamyl. Unsaturated compounds in the gas were very low and may have been polymerised or hydrogenated. The 68.5 per cent. of hydrogen in the gas may have originated from the isopropyl group and condensation of the aromatic nuclei. Small amounts of toluol and xylol were formed, but no benzol, the result probably of the great stability of the methyl group. Tetralin, representing the substituted aromatics, was cracked at 450° and 50–100 atm., for 19 hrs. 40 mins. A study of the products showed that cracking had taken three directions: (*a*) splitting off the hydrogenated side chains, with the formation of 55 per cent. of benzene homologs, (*b*) dehydrogenation of side chains and the formation of 30 per cent. of naphthaline,

(c) formation of 5 per cent. of condensation products. Six membered rings not only split off the side chains, but are dehydrogenated to aromatics, while five membered naphthenes are not dehydrogenated. Six membered naphthenes are practically absent in cracked gasoline. With regard to the thermodynamic principle underlying the observed cracking phenomena, the equilibrium constant may be calculated according to Nernst by the equation: log.K = the heat effect of the reaction ÷ 4.57Tabs.—the algebraic sum of the number of molecules taking part in the reaction × 1.75 × log.Tabs.—the sum of the "chemical constants." From the data of Nernst, 1.6 is taken as the chemical constant of hydrogen, 2.5 that of methane and 3.0 for all other hydrocarbons. After calculating log.K for various conceivable reactions for the cracking of paraffin hydrocarbons, using the heat of combustion as the heat effect of the reaction, the authors infer that that reaction will occur which gives the higher value for log.K at 477°. A tables of equations is given showing the possible break-up of aromatics with side chains, with log.K at 477° calculated for each case. The high values for log.K in the reactions in which styrol is a product of the decomposition are in harmony with the readiness with which that compound is known to be formed. The value of log.K for the dehydrogenation of cyclohexane at 27° is 21.8, that is, the reverse reaction will take place at this temperature.

At 327° log.K is O, and therefore K = 1. At equilibrium, cyclohexane and benzene are present in equal amounts, and Sabatier found that hydrogenation to cyclohexane took place up to 300°, but that above that temperature dehydrogenation occurred. Aromatics formed in cracking will condense with themselves to form diphenyl derivatives or polycyclic compounds, or will condense with unsaturated bodies to form polycyclics like anthracene and phenanthrene. Paraffins are almost incapable of entering into such condensations. W. H. T.

668. Recent Development in the Art of Cracking in the Vapour Phase. C. R. Wagner. *J. Inst. Petr. Techn.*, 1929, **15**, 484–492.

In 1809 Dalton experimented on the decomposition of hydrocarbon vapours by electric sparks, and sixteen years later Faraday isolated benzene and several of the unsaturated compounds. In 1855 Silliman made "oil gas" by high temperature cracking, and Berthelot's work on petroleum decomposition at high temperatures was published before 1870. Thorpe and Young began their work in 1865, and in 1862 Breitenlohner won a prize for a lamp oil produced by a high temperature vapour phase cracking process. Apparently, Cowper-Coles, in 1906, was the first to operate a cracking process to produce water fuel and his lead was rapidly followed by many others, all working in the vapour phase. The advent of the Burton liquid phase process changed the course of events and several processes made headway during the war. The chief stumbling block was excessive coke and gas formation. Another objection was high fuel consumption.

To-day, the "Gyro" vapour-phase process has plants to the capacity of 15,000 bbls. of gas oil per day working.

The refining of cracked spirit has always been difficult, but the difficulties have now been overcome. The smell is now quite pleasant, colouring matter is easily removed, and the gum problem has been solved. The motor fuel contains only traces of paraffins, being chiefly composed of olefines, aromatics, and naphthenes. Vapour-phase distillates are relatively low in sulphur content.

The fixed gas produced has a sp. gr. of about 1 and a fuel value of about 1700 B.Th.U. per cu. ft. It can be used as an enriching agent in gas manufacture, and owing to its ethylene content (about 27 per cent.) has great possibilities in the field of chemical manufacture. G. S.

669. Cracking of Natural Gas. *Petr. Times*, 1929, **21**, (546), 1160.

This article briefly reviews a paper read by J. A. Yunker at the May Convention of the Natural Gas Department of the American Gas Association. The plant consists of standard equipment as used for the production of blue water gas. The gaseous product obtained consists of about 90 per cent. hydrogen and less than 1 per cent. of hydrocarbon gases. Carbon-black, on recovery, amounts to 7 lbs. per 1000 cu. ft. of natural gas. The process differs only from water gas production, in which a bed of coke in the generator is brought to high temperature by an air blast followed by steam, in that natural gas is used at this stage instead of steam and, like the steam, undergoes dissociation in its passage. W. L.

670. Cracking Oil in the Liquid Phase. J. C. Black. U.S.P. 1,720,070. July 9, 1929. Appl. Oct. 26, 1926.

This is a process for cracking oil which comprises heating the oil in a confined stream under superatmospheric pressure to any predetermined cracking temperature. The temperature of the oil is measured by a number of pyrometers arranged along the stream. During the whole operation, oil is injected into the stream at all or any points where fluctuations of temperature, as indicated by the pyrometers, are observed. These fluctuations are due to the partial vaporisation of the oil stream, and by the injection of oil at these points the oil is maintained in its liquid phase throughout its course through the coils and subjected for a greater length of time to cracking in the liquid phase at the chosen temperature. The injected oil may be relatively cool oil or may be preheated to the cracking temperature of the coil. W. L.

671. Treating Hydrocarbons. J. C. Clancy. U.S.P. 1,721,728, July 23, 1929. Appl. Aug. 10, 1926.

Oils are desulphurised, prior to cracking in the presence of catalysts which are destroyed by sulphur, by treatment with fused sodium cyanide. Sodium thio-cyanate is formed in the reaction. The fusing point of the sodium cyanide may be lowered by the addition of sodium thiocyanate or formate to 200–350° C. Continued heating in the presence of the cyanide and thiocyanate cracks the oil. The oil may be treated either in the liquid or vapour phase. T. A. S.

672. Refining Petroleum Oil. R. Cross. U.S.P. 1,718,218. June 25, 1929. Appl. Dec. 17, 1924.

This invention relates to the treatment of cracking stock prior to its introduction to the cracking process and aims at the production of a gasoline having a low sulphur content and water-white colour.

The cracking stock is first treated with sodium plumbite solution in sufficient quantity to remove hydrogen sulphide and mercaptans as well as free sulphur. The oil is now washed with alkaline chlorinated water, settled and rapidly washed with 66° Bé sulphuric acid. The sludge is allowed to settle and the residual acid is neutralized with lime. It is claimed that gasoline produced from treated stock is water white in colour and has a sulphur content of about 0.1 per cent., and that it may be finished by treatment with sodium plumbite solution and clay filtration. W. L.

673. Method of Hydrocarbon-oil Conversion. G. Egloff. U.S.P. 1,705,181. Mar. 12, 1929. Appl. Dec. 4, 1925.

This invention relates to a method of hydrocarbon-oil conversion by means of heat and pressure, by which maximum yields of gasoline are obtained.

Fuel oil and kerosine-like bodies (such as pressure distillate bottoms) are heated simultaneously in separate heaters, and discharged together into a common expansion chamber and dephlegmator, the reflux from which is returned through another heater to the same expansion chamber. The whole system is maintained at a pressure much above atmospheric, and the temperature and pressure in each heating unit are controlled separately to avoid over-cracking of any one charging stock. Kerosine bodies are heated to a much higher temperature than fuel oil; the former then assist in the cracking of the fuel oil when mixing takes place in the expansion chamber.

D. G. P.

674. Catalytic Cracking of Hydrocarbons. G. Egloff, Assr. to Universal Oil Products Co. U.S.P. 1,722,042. July 23, 1929. Appl. Jan. 7, 1921.

Oil is heated, under 1000 lb. pressure, and passed into an expansion chamber in the upper portion of which is placed a tray containing catalytic material such as MCl_2 or CaO. After passing through the catalytic material, the vapours are passed to a condenser and receiver. For some refractory oils, such as Pine Island gas oil, a high pressure is maintained throughout the whole system, but for others, differential pressures are used, *e.g.* cracking coil 1000 lb., expansion chamber 300 lb., condenser 150 lb. and receiver 75 lb.

T. A. S.

675. Treating Hydrocarbons. G. Egloff, Assr. to Universal Oil Products Co. U.S.P. 1,722,043. July 23, 1929. Appl. Nov. 14, 1921.

Oil is distilled in a cracking still, the vapours passing to a dephlegmator from which the heavier fractions are returned to the still. The residual contents in the still are continuously being withdrawn, mixed with the oil returned from the dephlegmator and passed through a heated mass of refining material disposed in a container situated in the upper part of the still. After removal of carbon by this medium the oil is returned to the still for further cracking.

T. A. S.

676. Cooling the Heating Coils of an Oil Cracking Apparatus. G. Egloff and H. P. Benner. U.S.P. 1,725,067. Aug. 20, 1929. Appl. Jan. 14, 1925.

The invention relates particularly to that type of apparatus in which the oil is fed through a heating coil, thence delivered to an expansion chamber wherein vaporization takes place. In the operation of a plant of this type it is necessary to shut down periodically for cleaning, and to do this the plant must be cooled. This is usually accomplished by pumping cold oil through the entire system, which takes a considerable time. In order to avoid this loss of time, a connection is made between the outlet end of the cracking coil and the expansion chamber. At the end of a run the cold oil is pumped through the coil and back into the supply tank without passing through the remainder of the plant.

R. P.

677. Cracking of Hydrocarbon Oils. G. W. Gray. U.S.P. 1,724,760. Aug. 13, 1929. Appl. Oct. 28, 1924.

In the apparatus illustrated, three stills are adapted to be maintained under successively increased cracking temperatures and pressures. Each still is heated externally and is connected to a dephlegmator from which vapour lines lead to condensers. The reflux oil from the first dephlegmator is pumped into the second still and the reflux from the second dephlegmator to the third still.

Oil such as gas oil is pumped into the first still of the series which may be operating at a temperature of 750° F. and 150 lb. pressure. The vapours pass into the dephlegmator and the gasoline fraction is collected whilst the condensed reflux oil of the nature of kerosine is pumped into the second still which may be maintained at a pressure of about 200 lb. and a temperature of 780° to 800° F. Similarly, the vapour evolved from the second still is refluxed and the reflux condensate is pumped into the third still where it is subjected to a temperature of 810° to 850° F. and the pressure may be approximately 300 lbs.

The number of cracking zones required to obtain the maximum quantity of gasoline depends largely on the character of the raw oil and on the degree of cracking which has taken place in the preceding stills. The number may therefore be increased according to requirements.

Thus, a portion of the vapour from any still is drawn off and condensed as gasoline and is subjected to no further treatment whilst the reflux condensate is subjected to further cracking at an increased temperature and pressure. As the stills are of the same size it may be necessary to introduce fresh oil of predetermined character to each still to augment the supply of reflux oil from the preceding section and in order to do this a crude oil still is provided with a vapour line to a series of fractional condensers, each of which is connected to one of the cracking stills. A pump in the connecting line charges the condensate into the still against the contained pressure. R. P.

678. Treating Hydrocarbon Oils. C. J. Greenstreet, Assr. to Gasoline Corpn. U.S.P. 1,718,913. June 25, 1929. Appl. Aug. 20, 1920.

Heavy oil and steam are passed, under pressure, through heated tubes, arranged in a setting so that direct heating is avoided. With this arrangement no local overheating takes place and the temperature is more easily controlled. The duration of heating is carefully controlled and on leaving the tubes, the oil and vapours are immediately cooled to prevent further cracking. The light vapours are removed and condensed. The quantity of steam supplied is about 15 per cent. of the oil cracked and the operation is carried out at 110 lb. pressure. Cracking of gas-oil is carried out at temperatures "somewhat below 1100° F." T. A. S.

679. Converting Heavy Combustible Oils into Light Combustible Oils. P. L. Hahn. E.P. 289,482. April 27, 1928. Conv. (Germany), April 30, 1927.

In Patent No. 235,625 liquid fuels were treated for the production of combustible gas and this process is modified to convert heavy oils into light oils. Preliminary vaporization is effected by passing the oil through a molten metal bath. A filling agent, in the form of superheated steam or its constituents, is then mixed with the vapours at temperatures of 350° to 600° C., and the light hydrocarbons are condensed in the reaction chamber. G. S.

680. Pyrogenetic Cracking Process. R. W. Hanna. U.S.P. 1,724,476. Aug. 13, 1929. Appl. Oct. 15, 1927.

When certain oils are subjected to cracking it is difficult to obtain a reasonably high yield of gasoline without forming excessive quantities of coke. In the ordinary cracking process oil is passed through a furnace into an enlarged chamber from which vaporization may or may not take place and from which residuum is withdrawn. The gravity of the liquid within the reaction zone of such processes is much lower than that of the feed oil and an attempt is usually made to retain the oil in this chamber for a long period of time. The formation of carbon and the formation of residuum containing a high proportion of sediment is dependent on the length of time that the oil is

maintained in the reaction zone as the cracking reaction appears to form firstly, low boiling constituents and a heavy residuum, and if the latter is maintained at cracking temperature for a long period of time it undergoes further decomposition principally with the formation of coke and gas, but without any substantial increase in the amount of gasoline. It is the object of this invention to provide a process in which a high yield of gasoline may be obtained without the formation of coke, and with the production of a liquid residue suitable for use as a commercial fuel of suitable sediment content. The formation of coke and sediment is maintained at a minimum by cracking for a comparatively short time and thereby discharging from the reaction zone the heavy residuum more rapidly after its formation. However, in order to obtain the proper amount of cracking in the reaction zone, provision is made in the process for bringing the oil to a high temperature so that the short reaction period may still result in the same production of gasoline as in the ordinary processes. This is accomplished by first heating the oil at a comparatively low pressure to a temperature at which the oil is brought only to the point of incipient cracking and the final heating sufficient to effect a rapid cracking reaction is accomplished by compressing the materials undergoing treatment.

Essentially the plant consists of a heating coil through which the raw oil is pumped and therein attains a temperature of about 800° F. From the heating zone, the oil passes into a separating chamber where the pressure is about 250 lbs. to the sq. in., and here the vapours formed separate from the liquid. The vapours and the liquid are separately pumped into a reaction chamber at about 600 lbs. pressure where they mix and the temperature due to compression rises to about 850° F.

The desired cracking in the reaction chamber is regulated by the time and operating conditions maintained in the reaction chamber.

The relative quantities of vapour and liquid in the reaction chamber are adjusted by the position of the outlet line leading to a dephlegmator which separates the product into gasoline, reflux oil and residue. The residue and gasoline are withdrawn from the system and the reflux oil is returned into the feed line to the heating coil. R. P.

381. Cracking Hydrocarbon Oils. E. C. Herthel. U.S.P. 1,721,779 and 1,721,780. July 23. 1929. Appl. Jan. 12, 1928.

A pressure still system comprises a battery of heating tubes vertically arranged in a heating furnace, a vaporizing still, circulating connections including a pump and a valve for closing the connection between the vaporizing drum and the circulating pump. A reflux tower is mounted above the still level with suitable vapour and reflux return connections. Raw oil is introduced into the reflux tower and a draw off pipe discharges the unvaporized residue from the vaporizing drum. The vapours pass through a condenser to a gas separator.

The still is initially filled with the circulating valve open. The pump is started and the furnace fired. Raw oil is forced through the bearings of the pump. When the pressure on the system reaches 125/150 lbs. the circulating valve is closed and the still brought to operating temperature and pressure. At about the same time raw oil is introduced into the reflux tower, the rate being regulated to maintain the desired temperature at the top of the tower. Before the circulating still charge becomes saturated with pitch the circulating valve is gradually closed, then the discharge of heavy unvaporized oil from the drum is started and regulated to maintain a constant level within the drum. Reflux oil from the tower is introduced into the pump suction and returned through the heating tubes along with the raw oil.

R. P.

682. Pressure-Relief System. H. G. Hill, Assr. to Texas Co. U.S.P. 1,725,455. Aug. 20, 1929. Appl. Nov. 1, 1923.

The invention provides, in combination with a high pressure still for inflammable fluids and a main relief valve actuated by a predetermined excessive pressure, an auxiliary valve arranged to supply non-inflammable vapour or steam to a mixing chamber into which the main relief valve discharges. The auxiliary valve is of the spool type, actuated by a spring-loaded piston so adjusted as to operate at a pressure relatively lower than the predetermined excessive pressure yet higher than the normal working pressure of the still. The mixing chamber is provided with means for deflecting the inflammable fluids into intimate contact with the non-inflammable fluid, and is surmounted by a plurality of vertical tubes around which a cooling liquid is circulated. Above these tubes is a conical hood provided with a funnel at its apex open to the atmosphere. Within the hood a conical deflector aids the separation of condensable constituents from the mixture condensate, being removed from an annular pocket at the base of the hood and from the lower mixing chamber.

The automatic means for supplying non-inflammable fluid becomes effective in advance of the escape of volatile vapours, thus preparing the apparatus for preventing fire by driving the air out of the mixing chamber, whilst the circulating cooling medium effectively limits the temperature of the escaping mixture. L. H. A.

683. Art of Cracking Hydrocarbon Oils. E. W. Isom. U.S.P. 1,722,147. July 23, 1929. Appl. May 21, 1927. 1,722,222. July 23, 1929. Appl. Apr. 1, 1927.

The process is particularly advantageous in connection with the simultaneous treatment of relatively heavy oils, such as gas or fuel oils, and of lighter oils, such as kerosene or rerun pressure distillate bottoms. In the cracking of lighter oils it is advantageous to use a higher temperature of cracking than is used in the treatment of heavy oils.

In this process a stream of light oil is raised to cracking temperature in a heating coil and the hot products are discharged directly into a body of heavy oil, also maintained at cracking temperature in a cylindrical still. Vapours are taken from the still and subjected successively to two refluxing operations. In the first, the vapours are cooled by indirect heat exchange with fresh heavy oil which, thus preheated, flows into the still. In the second reflux chamber the vapours are further cooled by indirect heat exchange with fresh light oil, and the reflux condensate from both towers along with fresh light oil is pumped through the heating coil. If necessary the still may be heated externally.

Thus the deposition of carbon on the heating tubes is avoided by circulating only the lighter and more refractory oil, which does not cause excessive carbon formation, and avoiding recirculation of unvaporized oil which has been once subjected to cracking conditions and accordingly contains asphaltic constituents. R. P.

684. Art of Cracking Hydrocarbon Oils. E. W. Isom. U.S.P. 1,722,223. July 23, 1929. Appl. May 21, 1927.

A body of oil, contained in a still, is maintained at cracking temperature by circulating oil from the still through heating tubes and back into the body of oil. During its passage through the heating tubes the oil is heated to cracking temperature. The still is maintained about two-thirds full of oil and circulation is produced by a pump in the circulating system.

Vapours are taken from the still and passed through successive dephlegmators thence to the condenser. The heavy reflux oil from the first dephlegmator is returned directly to the still and the light reflux oil from the second dephlegmator is pumped through a second cracking coil situated in a separate furnace. The hot products from this coil are discharged into the circulating oil on its way to the first heating tubes. The injection of the hot oil may be used for promoting circulation.

Fresh oil may be added to the circulating oil or forced through the second cracking coil with the reflux. Light and heavy raw stock may thus be processed simultaneously and relatively refractory stocks such as kerosine or reflux condensate may be subjected to more severe cracking conditions than the heavier circulating oil in the still system. No external heat is applied to the still.

In one way of operating cracking may be carried out in the vapour phase in the second cracking coil and the vapours heated to a high temperature whilst the circulating oil is maintained at a relatively low temperature. The hot vapour-phase cracked products are reduced in temperature when injected into the circulating oil and undesirable reactions common to vapour phase processes are avoided. R. P.

685. Conversion of Hydrocarbons of High B.P. into others of Low B.P. J. Y. Johnson, Assr. to I. G. Farbenindustrie A.-G. E.P. 315,991. July 25, 1929. Appl. July 7, 1928.

Mineral oils, coal tars, etc., are converted into hydrocarbons of low b.p range in good yields by subjecting them to the action of a catalyst at temperatures of 400-600°C., at atmospheric or, preferably, elevated pressures, with or without such gases as H_2, N_2, CO, or water vapour. The catalysts employed are fundamentally aldehydes or ketones, with or without the addition of metals, or metallic oxides or salts, or such salts as are capable of adding on aldehydes or ketones, or the addition products so formed.

For example, producer tar freed from benzine is treated with 5 per cent by wt. of propionic aldehyde in an autoclave for 4 hrs. at 400/430° C. Of the cracked products 38 per cent. by wt. on the original boil up to 200° C.

Mineral oil freed from benzine is distilled with 5 per cent. by wt. of benzophenone, in a retort lined with Chromium-nickel steel, and 17 per cent. by wt. boiling up to 200° C., is produced.

Low-temperature tar is allowed to drop at about 600°C. in a stream of H_2 on to pumice impregnated with 5 per cent. of sodium formaldehyde sulphoxylate, contained in a copper-nickel steel column. 96 per cent. of liquid products are obtained, 29 per cent. on the original boiling up to 200° C.

Thin tar is treated with 4 per cent. by wt. of formalin and 1 per cent. of aluminium chloride in an autoclave for 3 hrs. at 400/440° C., 44.4 per cent. on the original boils up to 200° C. L. H. A.

686. Treating Hydrocarbons. W. J. Knox, Assr. to Petroleum Conversion Corpn. U.S.P. 1,715,239. May 28, 1929. Appl. Sept. 9, 1922.

Oil is vaporised and partially cracked on hot brickwork contained in a steel shell and used in a regenerative manner. The vapours are passed into a primary cracking chamber where they are brought into contact with oil which is kept circulating over packing material by means of a gas-lift worked with hot gases. From the primary cracking chamber, the gases are passed to a second packed chamber, maintained at cracking temperature, and where further cracking and the deposition of solid and liquid matter takes place. From the second chamber, the vapours pass to rectifying columns and condensers. The plant works at 250 lb. pressure. T. A. S.

687. Distillation and Cracking of Hydrocarbons, particularly Mineral Oils and Tars. H. Magnus. E.P. 287,525. June 14, 1929. Appl. Mar. 14, 1928. Conv. (Ger.), Mar. 23, 1927.

Mineral oil or tar is cracked under pressure and at elevated temperatures and the vapours arising in the process are withdrawn and fractionated. Fractions which it is desired to retain are collected and the remainder is passed on for further treatment under higher temperature and pressure, so that, in effect, the process is a multi-stage one which operates continuously. The liquid products arising in the pressure vessels are also continuously withdrawn, fractionated, and the unwanted fractions returned for further treatment. The changes of pressure in the cracking process are utilized for the generation of energy. The gases and vapours flow into a turbine at the pressure of the reaction vessel and thereupon expand. From here they pass through a heat exchanger to the fractionating condensers. The residual gas is forced back into the reaction chamber by a conveying device which is actuated by the turbine. The liquid constituents flow through a similar system. The turbines and conveyors are fitted with auxiliary steam lines controlled by automatic valves which interrupt the steam supply when the pressure in the reaction vessel has become high enough for operation.

W. L.

688. Distillation and Cracking of Hydrocarbons, particularly Mineral Oils and Tar. H. Magnus. E.P. 313,937. June 14, 1929. Appl. Mar. 14, 1928. Conv. (Ger.), Mar. 23, 1927.

This patent deals mainly with the construction of reaction vessels. It is claimed that the deposition of solid matter on the walls and bottom of the chamber and the formation of coke are avoided by making the heated walls of the vessel larger toward that side where heat is emitted than on the side where heat is taken up, thus keeping the temperature as low as possible. This is attained by constructing the heating walls with corrugations, ribs, fins, etc. The reaction vessel is partly filled with molten metal and the preheated raw material is injected through nozzles, arranged in a tangential direction, in the body of the molten metal. Circulating channels are provided in the walls of the vessel and suitable covers to these keep the metal in both rotary and vertical circulation, thus assuring that the raw material is heated uniformly and quickly without coming into direct contact with the walls of the vessel.

W. L.

689. Electric Arc Treatment of Liquid Hydrocarbons. Y. Mercier E.P. 316,336. July 22, 1929. Appl. April 20, 1928.

The invention claims improvements in the production of hydrogen and acetylene from petroleum oils, etc., by means of the electric arc. After striking the arc in the liquid the electrodes are separated to the maximum extent consistent with stability, thereby giving maximum gaseous output per kilowatt hour.

L. H. A.

690. Method of Cracking Hydrocarbons. T. Midgley Jr. U.S.P. 1,721,523. July 23, 1929. Appl. May 5, 1923.

The principal objects of the process are to increase the yield of aromatic hydrocarbons and to employ a non-hydrogenating catalyst.

The cracking chamber consists of a vertical cylindrical vessel, electrically heated, and covered with an insulating jacket. It is packed with pumice or other porous material which has been coated with a finely powdered alloy of iron and selenium. The operating temperature is 500° C. Kerosene is sup-

plied through the feed pipe and passes through the catalyst in the vapour phase. The kerosene cracks more readily in the presence of selenium than without and yields a high percentage of aromatic hydrocarbons. Yields as high as 40 per cent. aromatics and 24 per cent. gasoline have been obtained

The process may be varied by using as a base for the selenium, another metal such as nickel, cobalt, or lead, or tellurium may be used in place of selenium. The latter, however, is preferred, as it is cheaper and the yields obtained are higher.

The catalysts function as non-hydrogenating catalysts, *i.e.*, a catalyst which does not hydrogenate unsaturated hydrocarbons. R. P.

691. Process for the Decomposition of Hydrocarbons into Hydrocarbons containing Fewer Carbon Atoms. A. J. Van Peski. E.P. 315,193. July 18, 1929. Appl., Jan. 18, 1928.

This invention relates to a process for the decomposition of hydrocarbons, into hydrocarbons containing fewer carbon atoms, without the use of hydrogen or gases capable of giving rise to hydrogen. The reaction is satisfactorily carried out and controlled, by operating in the presence of metallic carbides, which under the conditions of the reaction, will form either more or less carbides without undue production of free carbon. The decomposition of the hydrocarbons may be effected at a pressure, either equal to, greater than or lower than atmospheric and either in the liquid or gaseous phase. The following examples illustrate the process :—

1. Propane is passed over granulated calcium carbide at temperatures ranging from 600° C. to 800° C. Decomposition into ethylene and methane takes place without deposition of carbon, while the catalyst retains its activity over a period of several days without showing any decrease.

2. Gas oil, having a boiling range of 220° C. to 340° C., is passed over calcium carbide at a temperature of 350° C. to 700° C. According to the temperature a smaller or larger amount of gas is formed, a considerable part of which consists of unsaturated hydrocarbons, containing fewer carbon atoms and a smaller or larger amount of liquid condensate having a boiling range of 60° C. to 180° C. H. E. T.

692. Decomposition of Hydrocarbons into Hydrocarbons containing Fewer Carbon Atoms. A. J. Van Peski. E.P. 315,800. July 18, 1929. Appl. Jan. 18, 1928.

Cracking is carried out in the absence of hydrogen (or gases giving rise thereto) but in the presence of one or more of the elements sulphur, antimony, arsenic, selenium or tellurium, or compounds of sulphur, antimony or arsenic, or mixtures of one or more of these elements and one or more compounds of these elements. The reaction may be effected in the presence of the substances mentioned together with one or more halogens or halogen compounds. W. S. N.

693. Apparatus for Treating Oils. R. T. Pollock, Assr. to Universal Oil Products Co. U.S.P. 1,721,973. July 23, 1929. Appl. Mar. 23, 1921.

The apparatus consists of a pair of chambers lined with heat-resisting material in each of which a cracking coil is mounted. Each chamber is fitted with air, steam and fuel lines and may be used either to receive oil which has been heated in the cracking coil contained in the companion chamber or as a combustion chamber to heat the cracking coil contained in it. The chambers are used alternately as expansion chambers or as furnaces, the fuel being the carbonaceous material which was deposited when used as an expansion chamber. T. A. S.

694. Oil Cracking: Means and Method. M. J. Trumble. U.S.P. 1,724,982. Aug. 20, 1929. Appl. Dec. 10, 1925.

A rotor similar to a toothed wheel revolves in the upper part of a furnace. The flame of the furnace impinges on the lower part of the rotor and a casing covers the upper part. Oil is preheated in the furnace flue then sprayed upon the upper part of the rotor. The vapours generated within the casing covering the rotor are conducted to a dephlegmator, from which the reflux oil returns to the rotor whilst the vapours pass to the condenser, preheating the raw oil in their course. To prevent flue gases passing into the cracking section and cracked vapours passing into the furnace, steam jets are placed at the points where the rotor leaves and enters the heating zone. R. P.

695. Hydrogenating and Cracking Hydrocarbons. M. J. Trumble. U.S.P. 1,725,320. Aug. 20, 1929. Appl., Dec. 24, 1924.

A horizontal still has two vertical hydrogenating chambers superimposed and is divided into upper and lower chambers. The still is fired from below and is provided in the lower chamber with a mechanical scraper in the form of an endless chain conveyer with blades attached.

This conveyer is drawn by a pair of sprocket wheels connected to a shaft which passes through the side, and near one end, of the still. At the other end of the still an adjustable rod attached to another pair of sprocket wheels extends through a gland and serves to tension the chain, compensate for wear, and overcome the difficulties incidental to the operation of a scraper in a chamber containing oil under pressure. The chain conveyer delivers oil and sediment to a pump which may communicate with the primary hydrogenator in such a manner that heavy uncracked residues may be blown into contact with a quantity of the hydrogenating agent.

Steam and sulphuric or hydrochloric acid are injected into the upper part of the primary hydrogenating chamber, where they are mixed with the heavy residue from the lower chamber of the still. This mixture comes into contact with the raw oil in the main section of the hydrogenator then the oil and hydrogenating agents enter the upper chamber of the still; sediment is removed by a spiral conveyer to carbon pots counter-current to the flow of oil and vapours which travel the length of the chamber and are discharged into the lower part of the still. A vapour outlet at the other end of this compartment conducts the oil into the secondary hydrogenator which contains a number of baffles and plates in order to favour a continuance of hydrogenation. The heavier oils collected in the secondary hydrogenator are returned to the primary chamber for further treatment whilst the gasoline vapours pass overhead and are condensed. R. P.

696. Method and Apparatus for Converting Hydrocarbons. L. J. Walsh. U.S.P. 1,725,434. Aug. 20, 1929. Appl. Nov. 28, 1923.

The method consists in heating a stream of oil to a cracking temperature under pressure, passing the hot stream into a vertical reaction chamber under pressure wherein the oil tends to form a lower layer of tar and an upper layer of lighter products. The latter are withdrawn continuously from near the top and the former from near the bottom, both being combined into a single stream, passed at reduced pressure into a vaporizing zone and the vapours condensed.

The plant consists of a heating coil and a vertical reaction chamber about 30 ft. high operating at about 250 lbs. pressure. The tar draw off pipe is in the form of an inverted U tube. The lighter products outlet is connected to the top of the U bend and extends about 2 ft. above it. Thus the tar and other products are mixed as they leave the reaction drum before entering the vaporizing drum where light oils and tar are again separated. R. P.

See also Abstracts Nos. 571, 588, 593, 595, 602, 635, 636, 639 and 724.

Oilfield Technology.

697. Underground Surveys of Oil Wells. A. Anderson. *Oil and Gas J.*, 1929, **27**, 49, 107, 166, 168, 170.

The surveying of adjacent wells showing great differences in production has shown that inclination has seriously diminished penetration of the oil zone. In making the surveys, a photographic recording mechanism was used on a string of drill-pipe or tubing, and it was possible to take 88 readings of inclination and direction in one run. Generally about 12 readings per 1,000 feet were taken per hour. Vertical angles were approximate to 10 minutes and direction to about 3°. Originally the instrument was designed to record readings up to 54° from the vertical, but was later modified to read to 65°.

In one case, where the baler could not be run to the bottom, the inclination was found to be 60°, and from cores obtained it was judged that the well reached a horizontal position.

In the examples quoted, the average loss in depth of wells finished between 3,000 and 4,000 ft. was 39.8 ft., and 38.4 ft. between 4,000 and 5,000 ft..

The drift is not necessarily an indication of the vertical depth, as this depends on the inclination and the depth at which the drift commences. In rotary holes the most interesting features are the moderate and continued bends which cause great drift and inclination. Such wells may appear perfect to the driller and give no indication of drift or curvature. A continuous curve has been observed amounting to 1.44 in. in a length of 40 ft., *i.e.*, an increase in inclination of 1° with each 30 ft. It is suggested that the drill-pipe tends to form a spiral which rotates like an attenuated corkscrew, and this would explain why the curvature in rotary wells is more moderate than if the pipe merely sagged to the lower side of an inclined hole.

Where the strata dips are less than 10° there is a tendency for the wells to butt into the structure, and a well may finish up several hundred feet higher on the structure than the derrick site would indicate. Where the dip is about 25° many wells follow the strike or take a general down dip direction.

Several interesting tables of results of well surveys are given showing, in one case, that at a measured depth of 5,379 ft. the vertical depth was 4,712 ft., the inclination at this depth being 48° and the drift from the vertical 2,100 ft.

G. S.

698. Problems of the Oklahoma City Pool. L. G. E. Bignell. *Oil and Gas J.*, 1929, **28**, 10, 54–55, 238.

An early problem in the Oklahoma City Pool was that of derrick foundations. The concrete mixture specified was 1 : 3 : 5 with 6 galls. water per sack of cement. Derricks are of the 122 ft. combination type.

All operations were carefully detailed before work commenced, and it was found that a lump sum figure for the complete contract was less than if all the work had been paid for as individual jobs. Drilling contracts required the hole to be kept within 5° from the vertical, and no rotary holes have been lost in this area to date. The handling of drill-pipe was found to be a heavier duty than landing casing. Assuming that 5,500 ft. of 6 in. 30 lb. drill-pipe is in use and that the mud has a gravity of 1.2, the total net load on the derrick would be 155,463 lbs. When "floating" in casing the mud is outside the casing only, and the total net load on the derrick when landing a 5,500 ft. string of 9 in. 40 lb. A.P.I. casing is only 37,757 lbs.

In one case, Petty No. 1 well, a gas pocket at 6,206 ft. blew up the tools, which became lodged at 500 ft. off the bottom. In fishing, the line was so

forced on the tools as to plug off the gas. When this plug was broken the gas again forced the second string up under the same conditions. A third attempt had a similar result, and a fourth fishing string is now in use. High-pressure gas pockets are characteristic of this area.

It is becoming usual practice to set casing as deep as possible, cement, and then bale mud until the string is clear. The casing is then checked for water-tightness and filled with water before further drilling. In some cases the gas pressure on piercing the cement plug has been sufficient to clear the well of water, while in others the balancing of gas and hydrostatic pressure has prevented the cable tools from working. In such cases it is necessary to shut down until the gas pressure has blown off through the water.

There are several large gas wells in the South Field with yields as high as 75 million cu. ft. per day, pressures in the producing horizons being as high as 2,500 lbs.

Kinter No. 1 well, which is producing under a back-pressure of 1,250 lbs., is making 1,400 to 1,700 bbls. of 67 to 71 A.P.I. gravity oil per day, 70 per cent. of which is heptane or heavier. With such high underground pressures it is possible to have such conditions of temperature and pressure that the gases, or at least the butane, propane, ethane, and some of the methane, would be reduced to liquids.

Future wells will have to be drilled with great care, and after they have passed the gas-lift stage of production the pumping will present many problems. Probably 3-in. tubing with 1 in. rods will be used, and with depths of 6,300 to 6,500 ft. the dead weight on the beam will be close to 40,000 lbs. To compensate stretch 72 in. stroke cranks will be used, and the speed will be governed by careful study. G. S.

699. Treating Oil by Use of Chemicals. R. T. Bright. *Oil and Gas J.*, 1929, **28**, 5, 40–80.

This article sets forth what is generally considered the best practice in breaking down the oil emulsion by the chemical method as the emulsion comes from the well. Chemical compounds which will resolve emulsions are classified into six groups according to their effect on the protective film which surrounds the finely divided particles of water. These groups are :—

1. Compounds which tend to take up water and form different substances.
2. Compounds that cause flocculation of the protective film.
3. Compounds that react with salts or organic acids present in the water.
4. Compounds that tend to form opposite types of emulsions.
5. Electolytes that tend to neutralize the charge on the surface of the emulsified water.
6. Solvents that tend to dissolve the protective film.

Substances that fall into Group 4 are considered the best for breaking down cut oil.

The layout and design of a general type of plant, which, with but few changes, can be adapted to most fields, is given diagrammatically.

The main points to be observed in the treatment are that the chemical should be introduced as close to the well as possible, where an oil and gas separator is in use, between the well and the separator, and should enter the flow line at a uniform rate.

Agitation should be reduced to a minimum after the emulsion enters the wash tank. The most favourable temperature for most oils is considered to be between 120 and 150° F., and a better and more uniform heat can be obtained, at a minimum cost, by running the emulsion through a boiler or heater, or by heating the wash tank with a circulating heated gas, than by the use of steam coils in the tank. W. L.

700. Conditions Encountered in Foreign Fields: Rumania. L. M. Drader. *J. Inst. Petr. Techn.*, 1929, **15**, 457–464.

Mining laws in Rumania stipulate that a detailed map, showing all derricks, buildings, etc., must be kept, together with accurate well logs and details of all operations. All engineers, managers, etc., must be fully qualified, and a number of staff positions must be held by Rumanian nationals.

Practically all drilling systems have been tried in Rumania, the majority of the wells having been drilled by the Canadian system. The Rotary method is becoming increasingly popular. The majority of the wells are baled and air and gas-lift are in the experimental stage only.

Transport is difficult, the greater part being carried out by oxen and horses.

Living conditions are comfortable, but the cost of living is high and prices fluctuate greatly.

Traces of oil are found from the Pliocene to the Lower Cretaceous, the commercial production being obtained almost entirely from the former.

G. S.

701. Re-Pressuring of Oil Sands. G. Elias. *J. Inst. Petr. Techn.*, 1929, **15**, 428–439.

It is estimated that the usual methods of production leave 70 to 80 per cent. of the oil in the sand and, of the solutions put forward for increasing ultimate production, those of the greatest possibilities involve driving the oil by the injection of water, gas or air into the sand. The water-drive has only been really successful in the Bradford field of Pennsylvania, but air and gas have had many successes. The gas, or air, drive has so developed that more gas is put in the sand during the flush production stage to maintain the pressure. The gas affects the oil in three ways—it reduces the viscosity, it carries the oil to low pressure areas, *i.e.*, the well bottom, and lifts the oil in the well.

The key well from the injection of gas should be within a group of wells intended to benefit, and should be in a good condition as regards tightness of casing.

Producing sands can also be used for gas storage during flush production by holding back pressure on the wells and by shutting in those wells which produce sufficient gas to neutralise the gas storage efforts. G. S.

702. Recovery Methods in Control of Water. R. V. A. Mills. *Oil and Oil Gas J.*, 1929, **27**, 48, 86, 89–90, 94, 96.

Water is best controlled by the conservation of as much natural gas pressure in the reservoir bed as is consistent with profitable working. Where the gas pressure is depleted it is necessary to consider what water control can be obtained by its restoration.

In fields such as the Appalachian, where gentle dips prevail, the first effect of re-pressuring is often to increase both oil and water production, the oil-water ratio remaining approximately the same. In the North Central Texas area good results from re-pressuring are reported, and it was found that the water was continually forced toward low-pressure areas in the sand. As a means of water control, the pressure should be maintained as uniform as possible through the whole productive area, and the value of early re-pressuring is emphasized.

The control of water by back pressure in air- or gas-lift operations has been successful in several fields. In the Panhandle field the water was reduced from 95 per cent. to 25 per cent. and oil production increased from 300 to 2,200 bbls. In some cases 5 lbs. reduction in pressure doubled the water

rate and decreased the oil rate. The best arrangement is to produce through the tubing, the bottom of which is raised to a point where the fluid head between its lowest point and the water surface, plus the head required to flow the oil, will exert sufficient pressure on the sand to prevent water coming up through the oil. Another method of holding back pressure on the sand is to restrict the flow from the well by an adjustable "bean."

In some areas it is profitable to produce as much water as possible with the oil to lower the water level in the productive formations. Oils of low viscosity are readily flushed into the wells by the water, whereas with oils of high viscosity the effect is often ruinous.

The effect of dewatering operations in an open-textured sand is that the oil drains downwards with the receding water and re-accumulates on the top of the remaining water.

Water encroachment in a large producing field may usually be traced to wells admitting top or bottom water to the formation, badly set casing being a common cause of top water admission. Abandoned wells which are improperly plugged are also a cause of serious water trouble.

The lowering of the water level in oil sands by dewatering operations is an economic as well as an engineering problem, but if the level in a number of test wells can be appreciably lowered, it may be inferred that there is a reasonable chance of success if the cost is not too great. G. S.

703. Oil Recovery by Natural Methods. H. P. Porter. *Oil and Gas. J.*, 1929, **28**, 13, 39, 75.

In order to assure the greatest ultimate recovery of oil by natural methods it is important to conserve gas and to utilize its many natural qualities. Natural gas should not be used for any other purpose until every consideration has been given to its use for recovery of oil. It reduces both the viscosity and surface tension of the oil and so helps to release it from the rock or sand.

Water pressure often plays an important part in oil recovery due to the flooding of the reservoir rock, whereby oil is forced along to the well.

Gravity also has an important bearing on the separation of oil, especially in pools where there is little or no gas or water pressure. Normally, the producer has to control all three forces so that he may utilize all of them to the best advantage, and has to determine when artificial methods of recovery can be employed economically. W. L.

704. The Disposal of Oil Field Brines. L. Schmidt and J. M. Devine. *Petr. Times*, 1929, **22**, (551), 207.

The disposal of brines which accompany oil production in various fields can be accomplished by (1) diversion into surface streams—a procedure to which objection is raised in some quarters due to the increased salinity which militates against its use as drinking water if more than 250 ppm. of chlorides and 1,000 pts. per million of total solids are present, and also that the increase in calcium and magnesium salts makes it too hard for many commercial purposes.

2. Evaporation ponds have been used with success, the brine, which is first freed from oil and B.S., being pumped into shallow ponds to which free access of wind and sun is available. Care should be taken that the brine does not drain into the upper strata upon which the supply of fresh water is dependent. This can often be prevented by building a seepage canal round the pond and pumping the seepage back on to the pond surface from time to time. The recovery of the salts thus obtained is not in many places a commercially profitable undertaking.

3. The brine may be returned to sub-surface formations by means of disused oil borings in isolated instances, but there is considerable difficulty to be expected in this connection both from the point of view of the rate of disposal which tends to decrease with increasing time due to clogging of the stratum to which the brine is being returned and also that this brine may endanger both the present and future oil supplies. A. R. S.

705. New Invention Designed to Show Hole's Deviation from Vertical. T. F. Smiley. *Oil and Gas J.*, 1929, **27**, 49, 44, 150–151.

M. M. Kinley has applied for a patent for an "Inclinometer" which may be placed in the drill-pipe immediately above the bit or may be lowered by cable into the well. The device is contained in a water-tight cylinder provided with a bale at the top and external threading at the lower end.

The recording unit is so arranged in its frame that the weights of the parts on one side of the axis are greater than on the other. Should the bit be deflected, the unit will swivel on pivots, so that the heavier side comes to rest at the lower side. A pendulum then swings inwardly and a pen outwardly to maintain contact with a recording strip. While the device is maintained in its inclined position a spring controlled motor causes the strip to move under the pen and so record a line corresponding to the inclination of the hole from the vertical. G. S.

706. Dehydrating Oilfield Emulsions by Chemical Reagents. *Petr. Times*, 1929, **553**, 301.

Many chemical reagents have a demulsifying action on oil-water emulsions, their efficiency being attributed to various properties. Thus sodium oleate or various sodium soaps, and sulphonates of certain organic acids, are soluble either in the oil or the water which accompanies it, this property of solubility having a marked influence on the interfacial relationship. Finely divided silica, iron oxide, and argillaceous materials also influence this interfacial relationship and bring about demulsification. Certain water soluble salts, such as sodium chloride, increase the interfacial tension with similar results.

Positively ionized electrolytes, such as sulphuric acid and ferric chloride, should neutralize the negative charges carried by the water droplets, thereby destroying the stability of the emulsion.

Flocculating agents, such as sodium chloride, ferrous sulphate, or sodium sulphate, should be useful where highly dispersable colloidal substances accumulated at the oil-water interface are functioning as emulsification agents.

Based on these theories a number of patented chemical reagents have been developed. "Tret-O-lite" has been prepared from several formulæ, an old one being 83 per cent. sodium oleate, 5.5 per cent. sodium resinate, 5 per cent. sodium silcate, 4 per cent. phenol, 1.5 per cent. paraffin, and 1 per cent. water. Recently 25 per cent. of liquid sulphonated oleic acid has been included in the formula, of which a 2 per cent. solution is made in water and added in small quantities to the emulsion, the temperature being raised if necessary. Another reagent, "Vez," is prepared according to 30 different formulæ, to meet various requirements.

With emulsions fresh from the well, the liquid reagent can be fed continuously into the lead lines in small quantity, separation then taking place in the storage tanks. When necessary, the latter are provided with steam coils, and the incoming emulsion is made to rise through a layer of hot water.

Installation costs for chemical treatment are low and operating costs moderate. The cost of the chemical reagent ranges from ¼ to 2¼ cents. per barrel; labour, power, heating, and evaporation losses bring the total cost to 3–17 cents. per barrel. L. H. A.

707. The Function of Natural Gas in the Production of Oil. *Petr. Times.* 1929, **22**, 550, 178.

This article is a survey of a report by H. C. Miller, which is being issued shortly by the U.S. Bureau of Mines.

The report is divided into two major parts, the first dealing with the importance of natural gas in the recovery of oil and giving descriptions of methods for controlling gas and oil production to obtain the greatest ultimate yield of oil. The second part summarizes the available facts with conclusions concerning the properties and action of oil and gas mixtures in underground formations.

The maintenance of back pressure on the face of the producing well is of primary importance, as the viscosity of the oil is kept at a lower point, the growth of gas bubbles, with a consequent increase in surface tension, is retarded, gas to oil ratios are less, and the ultimate recovery of oil is greater. To be most effective back pressure control should be co-operative throughout the pool.

Gas injection should begin in the early stages of oilfield development. The use of air for repressuring is not considered as desirable as gas, air being less soluble in oil than natural gas. Again, it does not reduce the viscosity or surface tension of the oil to the same degree as does gas, and where the oil is asphaltic or contains many unsaturated compounds, oxidation may take place causing operating difficulties.

The returned air-gas mixture is often explosive and unsuited for fuel purposes. W. L.

708. Recovery of Oil by Air and Gas Lift. *Oil and Gas J.*, 1929, **27**, 48, 31, 154–155.

The air-lift principle of raising water was discovered by Loscher in 1797, but it was not until 1864 that practical application was made of it when Crockford attempted to apply it to a Pennsylvanian oil well. In 1886 Poble patented, and successfully operated, the method in lifting water, and in the late nineties it was installed in several oilfields. It was again revived in 1920, and by 1925 its use was considerable.

The energy operating the lift is that contained in a volume of compressed gas released in bubble form in a fluid column at or near the submerged end of the discharge pipe. The driving force is the unbalanced hydrostatic pressure in the discharge pipe resulting from a fluid and gas column lighter than the outside opposed column of fluid.

The gas lift provides a means of increasing the rate of production and also for maintaining back-pressure and thereby controlling the gas-oil ratio. The gas-lift can lift fluid at a faster rate than any other means and can also handle fluids containing large amounts of suspended sand. The absence of moving parts is an advantage and fewer interruptions are occasioned in service than when using other methods.

Where gasoline extraction plant is in use, a greater volume of often richer gas is provided and a higher yield of gasoline secured. Recycling of the gas is also possible, leading to maximum economy.

As regards lifting effect, air and gas have no essential differences. It has been suggested that air, owing to its lower solubility in the oil, is preferable, but the use of air results in the gas produced with the oil being contaminated and the possibility of explosive mixtures being formed. Saline waters or certain sulphur compounds may, on contact with the air, become corrosive and attack tubing, etc.

The general action of the applied lift is that flow is induced or assisted by forcing compressed air or gas under pressure through tubing or through the annular space between the tubing and the casing. This air or gas "aerates" the oil column and by expansion carries the oil to the surface.

In starting the lift, a pressure due to "static" submergence" is met and decreases until the point of "working" submergence is reached, this being the combined forces acting downward at the foot of the eductor and equal to a column of oil sufficient to balance these forces. The operating pressure at the foot of the eductor is proportional to the working submergence and is expressed in lb./sq. in. as

Operating pressure = submergence in feet $\times$ 0.434 $\times$ sp. gr. of fluid.

The overall operating pressure at the well head includes the frictional pressure of gas delivery to the eductor foot, thus—

Overall operating pressure = operating pressure + delivery friction.

G. S.

709. Treating Emulsified Oil. G. Egloff. Assr. to Universal Oil Co. U.S.P. 1,722,041. July 23, 1929. Appl. Sept. 1, 1920.

Oil-water emulsions are broken by distillation under reduced pressure. The distillation is carried out in a number of stills arranged above one another in a combustion chamber. Heating is carried out by longitudinal burners arranged so that the liquid is heated near the surface, the oil in the bottom of the stills remaining cool. As distillation proceeds and the oil level is lowered the upper burners are extinguished and the lower burners lighted. In this way frothing is prevented. T. A. S.

710. Process of Treating Emulsions. C. Fischer and W. T. Reddish. U.S.P. 1,727,164. Sept. 3, 1929. Appl., Sept. 7, 1926.

This invention relates to the art of decomposing oil and water emulsions, and is particularly directed towards effecting the coalescence or stratification of the components of the emulsion. The accelerator of coalescence is an oil soluble bituminous or semi-bituminous substance such as pitch, animal, vegetable or mineral, having for preference a melting point between 35° and 95° C. The bituminous substance gives good results when used with mineral oil sulphonates thinned down with alcohol. H. E. T.

711. Process of Reclaiming Oil. C. Fischer and W. T. Reddish. U.S.P. 1,727,165. Sept. 3, 1929. Appl., Oct. 30, 1926.

A process is described for reclaiming oil from oil and water emulsions or from other media in which the oil may be dispersed. The oil and water emulsions are broken by altering the surface tension of the oil component of the emulsion. The surface tension depressant is an oil soluble mineral oil-sulphonic acid salt, while the preferred accelerator of coalescence of the oil drops is an oil soluble cohesive substance such as bitumen. Sodium aluminium sulphonate decomposes oil-water emulsions when introduced in the proportions of one part of sulphonic acid salt to one thousand parts of emulsion, the temperature being maintained at 150°-200° F. A reagent consisting of 85.95 per cent. of sodium aluminium sulphonate and 15.5 per cent. of bitumen, having a melting point between 35° and 95° C., is found to be most effective for collecting oil particles, and can easily be fed in aqueous solution or thinned with alcohol. H. E. T.

See also Abstract No. 568.

Geology.

712. GEOPHYSICAL METHODS OF PROSPECTING. (Introduction). G. A. Boutry. *La Revue Pétrolifère*. June, 1929, **325**, 821–825.

This paper gives an introductory account of the geophysical methods employed during oil prospection. The various hypotheses put forward to explain the origin of petroleum are first given, and these are followed by an account of the main petroliferous regions. The author then points out that geological methods of prospecting are by themselves hardly satisfactory as regards giving correct indications of the presence and extent of oil deposits. The problem of geophysical prospecting is next discussed, the various methods used being :—

1. Research of the variation of gravity, by the pendulum method, and the torsion balance.
2. Research on magnetic variations.
3. Research on electrical variations by means of continuous and alternating currents (conductibility, electro-chemical effects, etc.).
4. Research on variations of elasticity.
5. Variations of the geothermal gradient. Propagation in rocks of waves of short wave length. Radio-activity of petroliferous ground.

The principle of geophysical methods is simple, but their application is extremely delicate. The calculations involved are long, and the interpretation of the results obtained demands a serious geological knowledge. H. E. T.

713. GEOPHYSICAL METHODS OF PROSPECTING. Part I. G. A. Boutry. *La Revue Pétrolefère*, June, 1929, **327**, 893–896.

In this article the author discusses methods of measuring the acceleration due to gravity, and the Newtonian potential at a point. It is shown that the most accurate method of measuring "g," namely, by Kater's reversible pendulum is not used for geophysical determinations, on account of its tedious nature and because in many cases only relative values of "g" at various points on the earth's surface are required.

The method used to determine "g" during geophysical work is known as the "Invariable Pendulum." The instrument consists of a pendulum of known geometrical shape whose moment of inertia can be easily calculated. In general, the oscillating mass consist of two truncated cones united at their bases, while the stem is a heavy cylindrical steel rod, the point of suspension being an agate knife edge. In practice three similar pendulums are employed, oscillating in planes making angles of 120° with each other and resting on a single bronze support. The modern apparatus oscillates through a few minutes of arc only, and is placed in a vacuum whose temperature is carefully studied during each experiment. The measure of the ratio of values of "g" at two points consists simply in determining the period of oscillation of the invariable pendulum when placed successively at each of the two points.

The apparatus of "Trellfall and Pollock" is based on a very ingenious principle. It consists of a quartz fibre 30 cms. long and 40 microns in diameter stretched horizontally. At its centre is placed a small gilded copper lever whose centre of gravity lies outside the fibre. The quartz fibre has one end rigidly fixed, while the other is capable of being moved by means of a micrometer screw. By measuring the extent of torsion produced at two placed on the earth's surface by the small lever, the relative value of "g" is easily determined.

The next piece of apparatus described is the "Geobarometer." It consists in principle of an ordinary syphon barometer whose reservoir is open to the air, while the space over the liquid in the closed limb is filled with nitrogen. The above three instruments, although used in geophysical work, do not play a very important role mainly on account of their feeble sensibility.

The paper concludes with a mathematical discussion of the application of Eotvös Torsion Balance, to the measurement of the second differential coefficients of the Newtonian Potential, since it has been shown that these coefficients bear a marked relation at any particular place, to the presence of disturbing masses. H. E. T.

714. Further Aspects of the Mountain Building Problem. A. J. Bull. Proc. Geol. Assoc. Vol. XL, 1929, p. 105.

The author refers to an earlier article (Proc. Geol. Assoc. XXXVIII., p. 145), in which he had reviewed the chief hypothesis as to the cause of folded mountains, being driven to the conclusion that not one of them could account for the ascertained facts.

The most striking geological phenomenon which the thermal contraction hypothesis and other hypotheses fail to explain is the large sedimentation-subsidence, of usually elongated form, which as it sinks is kept filled with shallow water deposits and which after long geological time is folded parallel to its length, the folded mass rising isostatically to form a mountain chain. It is this long period of quiescence, with sinking areas and shallow transgressional seas flooding the continents, followed by a shorter period of local compression that so far has had no explanation to fit all the facts.

The usual way of attacking the problem has been to take some source of energy, compression due to the cooling of the earth, the force due to a continent sliding under gravity, the slowing of the earth's period of rotation or the force of isostatic adjustment, and then argue that the force in question has folded the rocks without considering whether existing tectonic structures could have been produced thereby. A better method may be to study the structures to find out what application of force could have produced them and, then, when it is known what forces are required, to consider what source of energy is possible.

Small scale experiments designed to imitate mountain structures have been tried by many geologists, the first definite result being obtained by Sir James Hall as long ago as 1815. He placed layers of cloth horizontally, loaded them with weighted boards, and subjected them to a horizontal compressive force. The resulting folds bore a close resemblance to the folds in the cliff sections of Berwickshire which he was studying.

Experiments of this type have been frequently repeated, but the solid base prevents the stresses being transmitted far into the material, the overfolds lack their upper limbs and nothing is indicated as to the mechanism of nappe-formation.

Before considering Alpine folding, where great thicknesses of the lithosphere are involved, it may be well to examine some cases in which relatively thin sheets of rock have moved bodily on their supports.

In the Jura Mountains the rocks are thrown into a series of symmetrical folds which are long dome-shaped structures arranged *en echelon*, the anticlines dying away and being replaced by synclines along their axes. The folded rocks consist of various formations down to the salt beds of the Middle Muschelkalk, the rocks below not being folded.

If a sheet of dough is allowed to slide down an inclined glass plate under the force of its own weight folds in *echelon* will be formed which show a close resemblance to some diagrams of the Jura.

It would appear that the Jura Mountains are the remains of a superficial sheet of rock which has been folded by simple compressional force, and that sliding under gravity played some part in this folding.

In the North-West Highlands a succession of great sheets of schist have moved over the rocks now occurring below them. In the Survey Memoirs a pressure from the E-S-E is postulated as the cause of the movement. It is,

however, significant that Cadell expresses the opinion that a force applied to a horizontal sheet would not be transmitted far, this opinion being the result of his study on the ground and of small scale experiments. If the land to the East stood high at the time of the movement, a constant gravity component would have been present to help any force applied from the East. The imbricate structures at the edges of the thrust sheets could have been produced by a sheet sliding bodily against the Torridonian and Lewisian as an obstacle. It would, therefore, appear that sliding under gravity played its part in this case also in producing the existing structure.

The outstanding problem of mountain building is the formation of a range of folded mountains consisting largely of sediments. The phenomenon may be cyclic, the initial stage being sedimentation in a sinking area such as the North Sea. The sedimentation-period is terminated by the formation of gentle folds, which become more and more pronounced, until finally a period of intense mashing together of the sediments supervenes, when most complex structures are formed, and the mass is raised above sea level and carved into mountains by denudation. The main cause both of the initiation and continuation of the subsidence and the subsequent folding must be looked for below the surface.

In the case of the Alps, the sediments which had accumulated in the Tethys Sea were first thrown into gentle anticlines, then overfolds were formed which travelled northwards in series after series of nappes, Palæozoic gneisses were forced into and upon the sediments, while granites of different ages were involved in the movements. Fine grained sediments of Liassic and later ages acted as lubricants between some of the great Pennine nappes, and were transformed into the Schistes Lustrés in the process. The southern extremities of the folded sedimentaries were tucked in under the still horizontal southern ends of the nappes, so that, with the inversion of the sediments in the lower portions of the nappes northward and these pli en retour in the southern region, a stratum that was originally laid down almost flat assumed finally the outline of a lop-sided mushroom.

The question arises as to what could have been the direction of the forces producing such a structure. A mere squeezing of the sediments would have produced fan-folding and then isoclinal folding with fracturing in the upper and more brittle layers. It is noteworthy that in the published descriptions of small scale experiments there has been no imitation of that long travel of recumbent folds that is the outstanding feature of Alpine structure.

In order to bring the extremities of a stratum once horizontal towards each other, the support on which it rests must contract, and this involves the removal of material downwards.

Nappe formation may be imitated in the following manner:—a series of layers of any material which is plastic under the conditions of the experiment are piled on each other, some powder or other substance which will prevent the layers from adhering too strongly being used. Two pieces of webbing are placed on a board with a slot cut in the middle, one end of each piece of webbing hanging down the slot. The pile of layers is placed on the webbing, the board tilted and weights hung on the ends of the webbing. As the webbing is slowly dragged down the lowest layers are dragged down with it, while the upper layers form anticlines which bend over and travel down the slope thus becoming nappes.

If the forces that produced the Alps were applied in the directions indicated in this experiment, one has to suppose that the gradual sinking, which permitted the sedimentation, was followed by a market movement of the sima downwards. At the same time the material coming in from the north and south dragged the sediments together. The piling up of light material would

cause high ground to be formed by isostatic adjustment, and the continued mashing together of these rocks would make the height more and more pronounced as the lithosphere thickened, and from this high ground nappes could descend as the folding continued. Should this have been the course of events in the Alps, then the under-drag from the North was greater than from the South. W. H. F.

715. Some Notes on Cretaceous Occurrences at Lizard Springs, Trinidad. P. W. Jarvis. *J. Inst. Petr. Techn.*, 1929, **15** 440–442.

Foraminiferal examination of about 250 samples collected at Lizard Springs showed a large number to contain Cretaceous fossils. Examination of the slides showed assemblages of from ten to twenty or more species, in most of which *Rzehakina epigona*, *Spiroplectoides clotho*, and *Nodosinella velascoensis* were found.

The *Rzehakina epigona* is one of the finest Upper Cretaceous markers in Trinidad, there being no difficulty in distinguishing it when suitably magnified.

The *Spiroplectoides clotho*, previously known as *S. annectens*, is also a good zone marker for the Upper Cretaceous, but the megalospheric and microscopic forms are different and might be mistaken for other species.

Nodosinella velascoensis is a good marker but has a descendent in the Eocene beds. It might also be taken for a *Nodosaria*, although close examination would show the difference.

The fossils of Lizard Springs correlate very closely with those of the Velasco States in Mexico. G. S.

716. Some Notes on a Portion of the Lizard Springs Anticline. R. H. Skelton. *J. Inst. Petr. Techn.*, 1929, **15**, 443–450.

The area dealt with is a rectangular block, the S.E. corner of which is approximately 10° 11 ft. 45 in. N. and 61° 4 ft. 21 in. W.

In the surface examination, over 200 samples were washed out and examined (see preceding abstract).

Five wells have been drilled and 136 core and bit samples collected and examined.

The deposits noted are of Tertiary (Lower Moruga, Lower Naparima, and St. Croix Formations) and Cretaceous (Silts and Clays) age.

The Lizard Springs Anticline is on the line of the Marac-Rock Dome Singinean uplifts and dips steeply to the north and south. The structure consists of a major truncated anticline, exposing a width of 1600 to 2500 ft. of pre-Moruga rocks, flanked on both sides by the Moruga formation. There is evidence of two distinct folding periods, one in Lower Middle Miocene times and one towards the end of the Pliocene. The anticline is cut into blocks by seven transverse faults.

If has been held by many that the Lizard Springs oil is of St. Croix age. The section shows that all the oil has been found in beds considered to be Cretaceous and which were also shattered. Either the oil is of Tertiary age and has only found a suitable reservoir in Cretaceous gault zones, or it is of Cretaceous origin. G. S.

717. Salt Structures in their Relation to Petroleum Deposits. A. F. v. Stahl. *Petr. Zeit.*, 1929, xxv., 948–949.

Although there are many recent references to the occurrences of petroleum near masses of rock salt, *e.g.*, Spindletop in Oklahama, Ochuiri in Roumania, Perm in the Urals, and German deposits, attention is called to the fact that a salt core or zone does not necessarily indicate the presence of oil. The presence of a salt core or zone is only a mechanical factor and has no direct

influence on the presence or absence of an oil-deposit. The author maintains that tectonic forces cause fracture of the petroliferous strata and the formation of anticlines. Large quantities of oil might accumulate under the salt zone but they would probably be of local extent since the surrounding strata would have been completely drained. However, these conclusions cannot be applied generally as the salt cores of Ochuiri are an exception. W. S. C.

718. The Gravitometer—A New Geophysical Instrument. B. D. Stevenson. *Oil and Gas J.*, 1929, 28, **9**, 154.

The design of the instrument is based on the well-known Crooks radiometer, which consists of a vacuum tube in which are mounted four square vanes on an upright needle. One side of each of the vanes is blackened in order to absorb light and heat waves, and the molecular action set up inside the tube causes the vanes to revolve. In measuring the extremely small anomalies of gravity, it is necessary to use an instrument whose forces are in the order of these anomalies themselves in order to obtain determinations of a sufficient magnitude to be recognised. If the temperature and light source are confined so that all other energies are excluded, it should show a difference in the speed of rotation whenever there is a change in gravity. The actual force manifested inside the vacuum tube is rather involved from a mathematical standpoint, but it has been determined that this force is approximately .02 of a dyne per square cm. one free mean path back from the edge of the vanes. The area thus being acted upon is very small and the reaction of the residual gas inside the vacuum tube, together with the friction on the needle point caused by the weight of the revolving system, constitutes the action absorbing the energy of the light stream. Whenever the light stream is in excess of these energies, the vane system revolves and if the light stream is held constant, the rate of rotation will remain constant provided there is no change in gravity. In actual field work, the gravitometer is set up at a datum station and the rate of rotation of the revolving system determined. The instrument is then moved to another station two tenths to three tenths of a mile distant and the rate of rotation again determined by a stop watch.

If the rate of rotation at the second station has increased, then the gravity at that point is less than at the datum station, and *vice versa*. Under ordinary conditions, a station can be made in 15 to 20 minutes, this including setting up, taking five observations and moving to the next station. The operation only requires the closest observation of the control of the electrical circuit, together with exact determination of rate of rotation. W. H. T.

Coal and Shale.

719. New Motor Fuel from Coal. *Petr. Times*, 1929, **21**, 546, 1162.

Among the papers read at the Second Bituminous Coal Conference held at Pittsburg, U.S.A., the section on Motor Coal and Steam Coal in the paper by J. G. Aerts, entitled "New Aspects of Coal Economy," strikes a new note. It discusses, first, the very large calorific losses in both steam and internal combustion engines, and then the liquefaction of coal by hydrogenation and low temperature carbonisation.

The production of pure elementary carbon of the alpha modification from coal is then described, and is believed to be a more logical idea of obtaining motor fuel from coal. Carbon dioxide is passed through a glowing bed of coal or coke and reduced to carbon monoxide. This in turn is decomposed by catalytic aid to give carbon and carbon dioxide. It is claimed that the solid carbon product named Carbonalpha is in molecular form, has a great affinity for oxygen, has a low combustion temperature (200° C.), has great

capillarity and hence great adsorptive capacity, the adsorbed substances becoming free again with a small rise in temperature and finally has great lubricating power.

Vapours distilled from coal are adsorbed by it forming a product which, it is claimed, can be used as a motor fuel in liquid, plastic or solid form. Mixed with heavy oils up to 20 to 30 per cent. it forms a fuel of easy ignition which will function perfectly in a motor. W. L.

720. Dry Distillation of Bituminous Shales, Brown Coal and the like. J. G. Grondel and C. L. Carlson. E.P. 316,483. Aug. 1, 1929. Appl. Oct. 29, 1928.

The apparatus consists of an elongated oven containing an endless chain grate composed of parallel sets of links. The grate is supported on rollers and is provided with upstanding edges to prevent the material fouling the walls of the oven. The grate moves over drums situated at each end of the oven. One of these drums has teeth provided on its circumference corresponding to the openings in the grate, and acts as the driving drum. On rotation, the teeth also act as cleaners to remove clogging slag and the like. Beneath this drum there is a scraper forming a part of the slag chute.

The heating device is situated in a chamber between the upper and lower parts of the grate. This chamber is open at the top and surrounds a channel for hot gases which are heated outside the oven. At the rear end of the oven, a hopper supplies coarse material and a second hopper, nearer the centre, feeds fine material which drops on top of the coarse material and is prevented by it from falling through the grate. In this way, both coarse and fine material can be processed in the same oven. R. P.

721. The Destructive Hydrogenation of Solid Carbonaceous Material. Imperial Chemical Industries. E.P. 314,213. June 27, 1929. Appl., June 22, 1928.

A process is described for the destructive hydrogenation of solid carbonaceous matter such as coal or lignite of a kind which on treatment at raised temperatures with hydrogen under pressures of the order of 200 atmospheres yields a viscous product. On acting on the said material with hydrogen under a pressure of at least 500 atmospheres, and at a temperature of 420° C., a mobile product is obtained. The coal or lignite can be treated in admixture with a suitable oil or tar acting as a vehicle. The mobile product produced has a ratio of oil to insoluble matter of 10 : 1, and the reaction time does not exceed 1 hour. W. H. T.

722. Destructive Hydrogenation of Carbonaceous Materials. A. J. V. Peski. E.P. 315,780. Jan. 18, 1928.

Carbonaceous materials are destructively hydrogenated at elevated temperatures at pressures below 50 atmospheres in the presence of catalysts consisting of one or more metallic carbides and/or one or more complex carbides.

For example, brown coal heated to 460° C. in the presence of 7·6 per cent. of a catalyst consisting substantially of nickel carbide with hydrogen at 45 atmospheres, yielded 4·9 per cent. kerosine, 3·7 per cent. cresols and 18·8 per cent. higher b.p. oil. Under similar conditions, but without a catalyst, the yield was 2·5 per cent. kerosine, 1·9 per cent. cresols and 4·0 per cent. high b.p. oil. G. S.

723. Eliminating Sulphur Compounds from Gases. J. Y. Johnson, Assr. to I. G. Farbenind. Akt. E.P., 310,063. April 22, 1929. Appl., Oct. 20, 1927.

The sulphur compounds contained in such gases as producer gas, water gas or coal gas are directly converted into the ammonium salts of the sulphur-oxygen acids by passing the gas at 150°—250° C. over solid active adsorbents together with sufficient oxygen or air to convert the sulphur compounds into sulphur oxides and adding the necessary amount of ammonia (if not already present in the gas) either before or after the catalytic treatment. The resulting ammonium salts are then separated from the gas by deposition in large chambers, by filtration, washing, electrical precipitation, etc. The contact material may be first charged with sulphur by passing the gas over it at the ordinary temperature together with only sufficient oxygen or air to oxidise the sulphur compounds to sulphur, and then oxygen or air alone or together with ammonia may be passed over the same at 150°–250° C. so that the sulphur is oxidised, the ammonium salts being deposited or washed out with aqueous ammonia. To avoid objectionable deposition of ammonium salts in inconvenient parts of the plant, the hot gases coming from the active adsorbent may be cooled at first only to such a degree that at least sufficient water vapour remains in the gas for the formation of ammonium salts (in particular, for the reaction:

$$SO_3+2NH_3+H_2O=(NH_4)_2\ SO_3)$$

and gaseous ammonia then added with thorough mixing.

Suitable contact materials are active charcoals, either alone or in compositions, *e.g.*, silica charcoal. Alloys or compounds of alkaline earth metals, of aluminium or of heavy metals with the non-metals of the 5th and 6th groups of the periodic system (other than oxygen) which are stable under the conditions employed, may also be used. Metals of the iron group, copper, and nitrides, phosphides or sulphides are mentioned as being particularly suitable. With these substances, as distinct from charcoals, temperatures up to 400° C. are employed.

Thus, brown-coal producer gas containing 14 gm. of sulphur per cubic metre is mixed with 10 per cent. by vol. of air and 2 per cent. by vol. of ammonia and passed at 250° C. over active charcoal. Producer gas containing 2.7 gm. of sulphur per cubic metre is mixed with 2 per cent. by vol. of air and passed over a granulated catalyst consisting of an alloy of 67 per cent. of iron, 22 per cent. of phosphorus and 11 per cent. of silicon at 400° C.; 0.4 per cent. by vol. of gaseous ammonia is added to the escaping gases at about 350° C. In either of these examples practically all the sulphur is oxidised. Producer gas containing 10 gm. of sulphur per cubic metre is mixed with 7–8 per cent. by vol. of air and passed at 400° C. over an iron-silicon-phosphorus alloy. The hot effluent gases are passed in counter-current heat exchange with fresh gases passing to the catalyst and are then cooled to 30° C. and mixed with 1.4—1.6 per cent. of gaseous ammonia. In all these examples the resulting ammonium salts are precipitated electrically. W. S. N.

724. Separation from Solid Residues of Oils Obtained in the Destructive Hydrogenation of Coals, Tars, Mineral Oils, Etc. J. Y. Johnson, Assr. to I. G. Farbendind. Akt. E.P., 312,657. May 21, 1929. Appl., Feb. 20, 1928.

Oils obtained by destructive hydrogenation of carbonaceous materials are separated from the solid residues by extraction with the aid of solvents which are recovered and used again. The solvent is a product of the hydrogenation which boils below 300° C. and is introduced into the treating vessel continuously together with the mixture. The removal of the extract and of the extracted residue is also effected continuously. The extraction can be

preceded by mechanical treatment or incomplete low temperature carbonisation, and the solid residue can be treated with different solvents in succession. The extracted residues are allowed to drop into a liquid exposed to the working pressure in order to separate completely the volatile carbonaceous products and especially the solvent contained in the residues. The surface tension of the liquid is lower than that of the carbonaceous product, and thus the residues can be discharged from the apparatus, preferably by passing them through a lock or the like. A suitable apparatus for carrying out the invention is described. W. H. T.

725. Production of Gaseous and Low Boiling Olefines and Diolefines from Bituminous Coal, Tars and Mineral Oils. J. V. Johnson (Comm. from I. G. Farb. Akt. Ges.). E.P. 313,756, June 23, 1928.

Granular bituminous coal, which is kept in motion by means of a current of vapours, is heated by means of an electric current. The vapours which pass over the coal are decomposed giving ethylene, propylene and butadiene. Instead of granular coal, broken firebrick, which has been soaked in iron salts to render it conductive, or granular metal may be used to decompose tar or oil vapours. The furnace is so arranged that the hot gases, after decomposition, preheat the vapours before they pass to the heating medium. A suitable furnace consists of a shaft 30 cms. square and 120 cms. high. Two graphite electrodes of 6 sq. decimetres in area are let into the walls. A temperature of about 650° C. is maintained with a current of 30 amps. at 380 volts. This will heat 40 kgs. per hour of tar vapours. From the vapours, light hydrocarbons are condensed and the residual gas has the composition. CO_2 and H_2S (1 per cent.), olefines (35 per cent.), CO (5 per cent.), H_2 (25 per cent.), CH_4 (23 per cent.) and N_2 (7 per cent.). The olefines fraction consists of about 20 per cent. butadiene. Powdered brown-coal is decomposed in a furnace of the size given at the rate of 90 kgs. per hour in a current of hydrogen circulated at the rate of 15 cu. metres per hour. The furnace is arranged so that the brown-coal is continuously fed to the furnace and the residue continuously removed. Decomposition is carried out at 700° C. and the residual gas contains 35 per cent. of olefines which are recovered by cooling or by activated charcoal. T. A. S.

726. Obtaining Liquid Products from Coal. M. Melamid. U.S.P. 1,723,431. Appl., Aug. 6, 1929, and Dec. 5, 1924.

The invention refers to a process of obtaining liquid products from coal or the like and is stated to be distinguished from prior processes of this kind by its simplicity and facility of operation.

The coal is heated with tin at a temperature not exceeding 600° C. in the presence of a stream of hydrogen, the comminuted solid coal being sprayed by the current of hydrogen into contact with the molten tin. W. H. T.

727. Separating Oil from Coal Tar. A Weindel, Assr. to Z. M. Stinnes. U.S.P. 1,722,872. Appl., July 30, 1929, and Dec. 12, 1924.

A process is described whereby certain lubricating materials present in tar may be isolated without being exposed to high temperatures which cause dissociation. The tar is mixed with a multiple quantity of petroleum benzine at ordinary temperature and the mixture afterwards allowed to stand at a lower temperature in order to separate out the tar oils from the precipitated asphaltic materials. The solvent is then removed from the purified oil by distillation, and the latter distilled up to 245° C. whereby the light oils are removed. W. H. T.

See also Abstracts Nos. 567, 685, 687 and 688.

ABSTRACTS.

Edited and arranged by W. H. THOMAS, A.R.S.M., and H. E. TESTER, B.Sc.

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General.

728. SURFACE TREATMENT OF GRAVEL AND MACADAM ROADS IN OREGON. R. H. Baldock. Eighth Ann. Asphalt Paving Congress. October and November, 1929.

High-speed pneumatic-tyred vehicles require roads with a smooth, hard, dustless surface and untreated gravel or broken stone surfaces are therefore unsuitable. The annual loss to owners of vehicles in operating over rough gravel and macadam surfaces in the form of loss of time, injury from dust, tyre wear and road shock is much greater than the loss to the highway authorities in increased maintenance costs. In Oregon, the first treatments were in the form of dust palliatives, using "fuel oil" to the extent of 6/10 of a gallon per square yard in two applications. These were not entirely successful, and, subsequently, heavy oil surface treatments were found to be more suitable. After sweeping, a prime coat ($\frac{1}{4}$–$\frac{1}{2}$ gal./sq. yd.) is given, followed by heavy oil ($\frac{1}{4}$–$\frac{1}{2}$ gal./sq. yd.) and a rolled dressing of clean screenings 0·01 cu. yds./sq. yd.). The oil (95 per cent. asphaltic oil, heavy tar or soft asphalt) is spread by a pressure distributer at 400° F., while the stone is laid by a special spreader box. In certain cases, the road is scarified to a depth of from 2–3 ins., and mixed with an oil (60–70 per cent. asphalt and 200–300 secs. visc. say purol) to the extent of $\frac{1}{2}$ gal. per sq. yd.

For bituminous macadam construction, the rock used is crushed and graded basalt together with Californian asphalt (95 per cent. asphalt, 225 secs. visc. at 122° F., or soft asphalt-penetration 150–250 at 25° C.). Emulsified asphalt has been used both for surface treatment and bituminous macadam. A home-made product is sometimes utilised, the water being applied at air temperature immediately ahead of the oil spray (20 lb. press. at 400° F.). Lye and soap jelly is dissolved in the water and the latter applied to the extent of 10 per cent. by vol. of the hot oil. For new water bound foundations and wearing course, a minimum thickness of 6 ins. of broken stone of $2\frac{1}{2}$ ins. maximum size is used.

Where surface treatment is contemplated, the foundation course is covered with a 2 ins. layer of $1\frac{1}{2}$ ins. stone and a 1 in. layer of $\frac{3}{4}$ in. stone to afford a smooth surface. With regard to a road mixed treatment, it is necessary to cover the foundation course with a 3 ins. layer of $\frac{3}{4}$ in. material. Fifty per cent. of the top material should pass a $\frac{1}{4}$ in. screen and 8 per cent. should

pass a 200-mesh screen. The author gives the costs of four distinct types of treatment, the cost of maintenance of the surface treated type and that of non-skid treatment for black pavement. W. H. T.

729. Asphaltic Concrete for Highways. G. W. Craig. *Highway Engineer and Contractor*, 1929, 35 (1), 48.

Asphaltic concrete base or "black base" should be made from suitable crushed stone, slag or gravel, with the addition of the proper amount of sand and asphaltic cement. Specifications should follow fairly closely the standard developed for binder course, save that for thick bases the maximum size of stone should be increased proportionately. Fine and coarse aggregates should be well graded from maximum to minimum. The same care in the preparation of the sub-grade is as essential as in any other type of base. When under construction laboratory and plant control are essential and close attention should be given to the temperature of the material when applied. Rolling should be continuous till the pavement is true to crown and grade and till it has cooled to about air temperature.

Asphaltic concrete base provides homogeneity of mass and a uniform contact with the sub-grade, and by its inherent flexibility the possibility of foundation rupturing is reduced to a minimum. It insures against moisture in the foundation structure and provides freedom from cracks and upheavals, also it is easily and cheaply repaired. W. L.

730. Merriman Asphalt Patch Plant. *Highway Eng. and Contractor*, 1929, 35 (1), 94.

The apparatus is a self-propelled machine for use in patching asphalt streets, etc. The material and the asphalt are carried in separate containers. When in operation the material is heated in a Merriman drum, screened, weighed and mixed with the requisite quantity of asphalt. The mixer is of the pug-mill type and the heating of the drum and asphalt kettle is by fuel oil burners. A tool heater is incorporated on one side of the plant. The total weight is 9800 lbs. and the power unit is a 20 h.p. petrol engine. The plant is operated entirely from one side and is automatic from one end to the other. W. L.

731. Effect of Aggregates on Stability of Bituminous Mixtures. P. Hubbard. A.S.T.M. Symposium on Mineral Aggregates. June 1929.

Stability is defined as resistance to deformation or internal displacement under such stresses as are imposed by traffic. Mineral aggregates may possess a considerable degree of inherent stability irrespective of the presence of bituminous cement. As they constitute the great bulk and weight of a bituminous mixture, their inherent stability becomes an important part of the stability of the mixture when it is properly designed. Thorough compaction is the primary factor affecting stability, and density of the compacted mass is of next importance. Softness of the mineral fragments may or may not be advantageous from the standpoint of stability depending upon the degree of softness and size of particles. Various classes of coarse and fine aggregates are discussed with reference to stability, and it is shown that the value of grading with relation to density is commonly over-estimated. In general, the addition of mineral filler will increase stability so long as it reduces voids in the aggregate, but when further addition begins to increase voids, then stability of the mixture also begins to decrease. Two sands of the same grading may possess entirely different stability values, probably due to difference in surface texture. Mineral fillers may also vary greatly in their stabilising values due to their differences in fineness and surface

texture. There are, so many factors affecting the stability of bituminous mixtures that it is impossible to indicate by reference to the usual tests to which the individual constituents are subjected what stability may be produced in mixtures in which they are combined in definite proportions.

W. H. T.

732. The Ceptura Oilfield of Roumania. *The Petr. Times*, XXII., 558, 569.

The Ceptura Oilfield has rapidly come to the front as one of the most important in Roumania, for the output of crude oil from this field represents one-sixth of the total production of the country.

The wells follow the crests of the ridges forming the valleys of Ceptura and Cricov, and the field takes the form of a Pliocene anticline which can be traced from Tartaru along the borders of the village of Malu-Rosu, past Suditi and Urloi towards the valley of the Cricov. On its productive side it falls at an angle of 14°, and the Levantine, Dacian, and Pontian formations crop out on the hillside, whereas the Meotic formation appears at the surface only at Tartaru and Dobruta, outside the limits of the Ceptura field. The oil occurs in the Meotic formation, which has been explored to a depth of 1300 ft. Two important oil sands are known to exist, the upper 120 to 260 ft. in thickness and the lower between 800 and 1700 ft.

The geological characteristics are exactly known, due mainly to close collaboration between the companies concerned, who have pooled their investigations for the common benefit. One advantage of the co-operation has led to accurate knowledge of water levels, which are dealt with so expeditiously that there is not a single watered well in the district.

The whole field has been developed according to plan and attention paid to the proper spacing of wells, the average distance maintained by each company in the same deposit being roughly 200 yards.

A great advance has been made in drilling technique by the introduction of the rotary system, whereby it is possible to drill a well in from 8 to 11 months instead of three years with the old Canadian system.

Fishtail bits of 12 ins. are employed and adequate steam power permits of rotation at the rate of 120 revolutions per minute. Drilling is particularly rapid in the upper Pontian strata. Holes, however, are apt to deviate from the vertical, but this is overcome largely by the use of pressure indicators, and every well in the Ceptura field is fitted with these instruments.

Efficient working has led to a reduction of costs per metre drilled from 13,746 lei in 1927 to 5,500 lei in 1929.

The crude oil yields 17 per cent. of petrol. It is a paraffin base oil congealing at 28° and each well must be fitted with paraffin cutters and pipe lines, and tanks must be heated to prevent the oil solidifying.

The history of the Ceptura oilfield proves what can be done by the application of modern methods of investigation, drilling and production, and by following a broad-minded policy in the general interest. It has reached its present state of efficiency and importance only by the close collaboration between the chief operating companies and pooling the data obtained from individual properties.

R. P.

733. Asphalt Road-Building Machine. *Petr. Zeit.*, 1929, 25, 1391–1395.

An efficient machine for road-building is described and diagrams given, viz., newly-constructed Millars' type. Such a machine is necessary for the production of road surfaces from asphalt macadam, *i.e.*, a mixture of bitumen, sand and stone chips. It is capable of mixing 10 tons of sand and asphalt in one hour. The procedure is as follows: The flux material, sand, and broken stone, or only sand, are drawn up by a bucket conveyer into a drying

drum which revolves slowly over a heating space. The hot, dry flux issuing from the drum outlet is drawn up by a second bucket conveyer into a revolving screen or into a storage reservoir. The latter fills and is brought to a lever scale over the mixing vessels. Hot bitumen is added by means of a movable metal-vessel by air pressure, and special weighing devices for flux and bitumen are fitted. A double mixer is present with a corresponding scoop system, according to whether the material to be treated is fine or coarse. The mixture so prepared is passed in a conveyer to the area of operation on the road.

The newly-constructed machine has all the service levers combined on a platform, so that one man can produce the ready mixture from the heated raw material without unnecessary motion. The high parts of the apparatus are easily accessible by ladders. Power can be applied by means of a tractor, roller, and suitable motor (20 h.p.). The machine is made of cast steel and can be driven and heated from both sides. The firing is arranged for solid or liquid fuels, and the mixings are carried out by weight, the fillers being added by special weighing devices. W. S. C.

734. Asphalt Surfaces for Bridge Floors. J. E. Pennybacker. *Highway Eng. and Contractor*, 1929 35 (1), 54.

Asphalt is especially suitable for bridge floors, because of its capacity for reducing vibration, eliminating noise and also because of its waterproofing qualities and comparatively light weight. Also such floors can be constructed rapidly and renewed at a minimum cost.

Mat coats, from $\frac{1}{4}$ to $\frac{3}{4}$ in. thick, as a protection for timber or concrete floors, are suitable for light traffic bridges but do not stand well under heavy traffic.

Asphalt pavements, 2 or 3 ins. thick, are among the best wearing surfaces for concrete bridge floors, when the items of cost, weight and surface are considered. The Delaware River Bridge is paved with sheet asphalt of $1\frac{1}{2}$ ins. binder and 1 in. wearing surface.

Specification for the binder course is :—

Coarse aggregate retained on a 10-mesh sieve	60–80%
Sand material passing a 10-mesh sieve	15–35%

Specification for the wearing course is :—

Retained on a 10-mesh sieve	0–2 %
Passing a 10-mesh, retained on a 40-mesh sieve	15–35%
,, 40-mesh, ,, ,, 80-mesh ,, ...	25–40%
,, 80-mesh, ,, ,, 200-mesh ,, ...	20–30%
,, 200-mesh sieve	14–18%
Asphalt	9½–11½%

Experience shows that the asphalt content of the surfacing course can be kept close to the lower limit.

Paved surfaces must be adequately supported from below, usually by reinforced cement concrete slabs, steel buckle plates or timber.

The irregularities in the surface presented by steel buckle plates should be levelled with an asphalt concrete mixture before the application of the surfacing course.

Wooden floors may be surfaced with most types of asphalt, but should have no movement under traffic.

Several examples of resurfacing worn floors are given with the methods of application used. W. L.

735. Bituminous Resurfacing of Idaho Gravel Roads. W. A. Scott. *Highway Eng. and Contractor*, 1929, 35 (2), 47.

The gravel and crushed rock roads, at the beginning of 1927, included a large mileage that required a kind of resurfacing that would meet the needs of increasing traffic. It was estimated that the surface losses, due to tyre traction and wind, amounted annually to nearly an inch in depth of the macadam material, or approximately 13 per cent. of the original surfacing. This depreciation led to the adoption of bituminous resurfacing as a phase of maintenance. It was begun in a limited way with the oil treatment of 74 miles in 1927 and 240 miles in 1928, and was limited to roads having a 6 ins. depth of gravel surfacing. The present practice is to scarify the macadam surface, the depth of scarification depending on the quantity of oil to be applied. The oil has an asphalt residue content of 60 to 70 per cent. and is heated to 160° F. As regards quantity, in some instances there are three spreads of ½ gal each per sq. yd., the pressure spreader covering half the roadway at each trip. Harrowing the surface before and after application serves to give the oil a uniform contact with the aggregate. The mixing is completed by a blade grader. The base intended to carry the 2 ins. bituminous course is required to be compacted and firm so that the blading after oiling will not take up any portion of the base material. The preparation of the base is made several months in advance of the oiling and is often finished with a light coating of oiled macadam. The cementing elements in the base consist of sand and screenings with a low percentage of clay. Material for the 2 ins. oil mat requires the spreading of crushed rock or gravel at the rate of 800 cu. yds. per mile. This material is graded to pass a 1 in. circular opening, 30–45 per cent. of which passes a ¼ in. screen.

W. H. T.

736. Fine Aggregate in Bituminous Mixtures. H. W. Skidmore. A.S.T.M. Symposium on Mineral Aggregates. June 1929.

The fine aggregate portion of bituminous mixtures may serve primarily as a void filler in coarse mixtures until the amount of fine material exceeds the coarse, whereupon the situation is reversed and the coarse aggregate reduces voids in the fine; or it may constitute the whole mineral structure. In all filled mixtures there are two distinct aggregates that require separate treatment in test and design: fine aggregate exclusive of filler (the normal sand portion), and with filler. They have many properties and functions in common, but also require separate consideration before they are combined. In the case of unfilled fine aggregate (sand) the important characteristics are voids in dry state, grading, specific gravity, porosity and rugosity, shape of grains, stabilising ability, hardness and toughness. In the case of filler, these characteristics are void-reducing ability, stabilising ability, fineness, specific gravity, shape of particles and porosity. No two aggregates or fillers behave exactly alike in mixtures, hence criteria cannot be established for a given kind or class. Composite aggregates exclusive of filler require preliminary examination to ascertain the optimum combination with respect to practicability, voids, grading and specific gravity. Filler must be added in varying amounts for further study and the mixtures finally tested for compressibility (mixture voids), stability (resistance to shear) at 140° F., absorption of moisture and denseness. Certain porous aggregates have distinct advantages over non-porous ones both with respect to stability and durability in service. Certain fillers may be excellent void reducers and yet produce poor compressibility of mixture, while others impart marked increase in stability.

W. H. T.

737. Latest Developments in Asphalt Paving Practice. H. W. Skidmore. *Roads and Street*, 1929, 69, 255.

Subgrade is so important that every operation depends upon its character, and fundamentally it should be considered before the base and surfacing are designed. Various means of subgrade treatment are :—

1. Porous layers on certain soils.

2. Insulating layers of fine granular material to prevent soft soils from working up into macadam bases.

3. Bituminous treatment to break up capilarity.

4. Compacted layers of gravel, stone, etc., to correct lack of uniformity of subgrade support.

5. Drainage to remove moisture and to prevent wetness by intercepting flow from water-bearing strata.

Traffic-bound bases, such as old road beds, are nearly always excellent foundations.

Black base is one of the most important developments of asphalt paving practice.

Asphalt surfaces are a great protection to portland cement concrete slabs but do not prevent cracks. Soft asphalt and rich mixtures are no cure for this evil.

Two developments in the field of asphalt paving mixtures are first rational mixture design from actual scientific basis, and, secondly, the stability or shear test for rating mixtures according to traffic requirements. This test has exposed the fallacy of attempting to increase stability by using harder asphalt cement and by laying dry mixtures containing less bitumen than will comfortably fill the voids in the mineral aggregate. Modern practice calls for two cardinal principles, use as much bitumen as the material will carry, and use a soft bitumen consistent with the traffic and the actual stability afforded by the mixture. Increase in stability, if needed, can be accomplished by manipulating the mineral aggregate, especially the filler. Thorough mixing of the aggregate before the asphalt cement is introduced is of primary importance. W. L.

738. Surface Treatment of Earth Roads in North Carolina. J. D. Waldrop. *Highway Eng. and Contractor*, 1929, 35 (2), 61. Paper presented before Seventh Annual Paving Conference, New Orleans.

Surface treatment of earth roads in North Carolina was begun in 1924, using a light oil, and in 1925 the AC-2 specification was developed. This specifies an asphaltic cement as follows :—

Penetration, base material, at 77° F.	150–180
Distillation, cut back as follows—	
Initial boiling point, not less than	60° C.
Distillate to 105° C., not more than	8%
,, 140° C., ,, ,, ...	30%
,, 200° C., ,, ,, ...	80%
Viscosity, finished product (Engler)	15–25

Asphalt had hitherto been used both as a prime coat and also for second and third applications, but trouble was encountered with peeling and breaking up, while cold weather caused scaling and permanent breaks. The AC-2 material demonstrated its ability to hold sand or pea gravel but showed decided tendency to either peel or push. It was noted that the asphaltic oil used lacked some qualification needed to cause it to adhere properly to the road and therefore tar was used for the prime coat. It was seen, however,

that the sand absorbed the tar which was supposed to penetrate the road itself, and thus there was no unification between the prime coat and the road. Later, an experiment was conducted with stone chips for cover material and tar as binder. The success of the experiment led to the discarding of sand and the adoption of stone for all new work except in those instances where the price of stone was prohibitive and where an excellent grade of coarse clean sand or gravel was available. Definite policies and methods of construction were adopted and may be briefly stated as follows :—

(1) Tar used almost entirely for prime coats.

(2) No cover material placed on this prime coat.

(3) Prime coat allowed to stand until maximum penetration into road has been secured, this period varying from 2 to 5 days.

(4) Traffic kept off prime coat.

(5) Where sand is used for second and third applications, asphalt is the bituminous binder material.

(6) Where stone chips are used for second application, tar is the bituminous binder material.

(7) Where asphalt and sand are used, the original construction calls for a prime coat and two seal coats.

(8) Where tar and stone are used, the original construction calls for a prime coat and a seal coat.

Under (8) above, the original construction is followed by an additional treatment of smaller stone and a cold application of tar the first season following the original work. W. H. T.

Chemistry of Petroleum.

739. Amyl Alcohols from Pentanes. E. E. Ayres. *Ind. Eng. Chem.*, vol. 21, 10, 899. October 1929.

At the present time amyl alcohols are being manufactured in a plant in which 22 tons of chlorine a day are being fed continuously into a 60-mile-per-hour stream of hot pentane vapour. One hundred thousand gallons of pentane pass through the reaction zone every day, but at any given instant only 3 gallons of pentane are present within the reaction zone, the reaction with chlorine being completed in about 2½ seconds, while only 8 ounces of chlorine are in contact with the pentane vapour at any given moment. A train of fractionating columns separate the reaction product into three components : (*a*) hydrogen chloride, which is absorbed in water, producing 33 tons of 21° Bé. hydrochloric acid per day ; (*b*) a small amount of polychloride residue ; and (*c*) pure amyl chlorides.

Hydrolysis of the amyl chloride is accomplished in a system of reservoirs, heaters and pumps through which a hot emulsion of amyl alcohol and water is circulated at 500 gallons a minute. Amyl chloride and aqueous caustic soda are continuously pumped into this circulating system, and two things are continuously removed : (*a*) a saturated salt solution which is returned to the electrolytic cells for the manufacture of chlorine and (*b*) a vapour containing mainly isomeric amyl alcohols. Three hundred gallons of amyl chloride are always present in the hydrolysis system, while 300 gallons are hydrolysed each hour. The vapours from hydrolysis are separated by a train of fractionating columns into five components : (*a*) amylchloride, which is returned to be hydrolysed ; (*b*) amylene which is later hydrated to the alcohols ; (*c*) diamyl ether ; (*d*) amyl alcohol fractions which are cut out

for special uses; and (*e*) the mixture of amyl alcohols, marketed under the trade name "Pentasol," for use in pyroxylin lacquers. The source of the pentanes from which the amyl alcohols are manufactured is natural gas.

The chemistry of the various reactions occurring in the plant is discussed in some detail. H. E. T.

740. A New Petroleum By-Product: Octane-Sulfone. E. L. Baldeschwieler. *J. Amer. Chem. Soc.*, 1929, **51**, 2969–2978.

A compound of empirical formula $C_8H_{16}SO_2$ has been obtained from one of the by-products of the manufacture of alcohols and separated as crystals of a high degree of purity. Its behaviour is strictly analogous to that of saltones in the aromatic and camphene series, and is undoubtedly an octane-sultone. On refluxing with alkalies, various alkaline salts are obtained whose different chemical properties are explained by a shifting of the double bond generated from the hydroxyl group. The compound is interesting to the petroleum technologist as it is volatile in steam and hence aliphatic sultones may be partly responsible for the sulphur content of refined gasolines. Attempts are now being made to synthesise sultones in the aliphatic series

W. H. T.

741. Action of Sulphuric Acid on Mercaptans. S. F. Birch and W. S. Norris. *Ind. Eng. Chem.*, 1929, **21**, **11**, 1087.

During the refining of light petroleum distillates by means of sulphuric acid the authors found that a minute quantity of a highly corrosive substance is produced, which is soluble in the spirit and renders it at the boiling point corrosive to copper. The corrosive substance is not soluble in concentrated sulphuric acid, and does not react in the cold with metallic mercury, which distinguishes it from elementary sulphur.

A systematic study was therefore carried out on the action of concentrated and fuming sulphuric acid on organic sulphur compounds in naphtha solution. The naphtha used was prepared from commercial painter's naphtha by distilling in a current of steam to a 5 per cent. residue; the distillate was sulphur-free, commenced to boil at 130° C., 90 per cent. distilling between 150° C. and 180° C., and it had a density of 0·777 at 15·5° C. When distilled to dryness in the presence of copper, this solvent did not tarnish the metal. The striking fact was discovered that only when the original solution contains a mercaptan was it corrosive at the boiling point to copper. The effect of introducing various mercaptans and sulphides into the initial sulphur-free naphtha was determined, and it was found that mercaptans alone of the organic compounds of sulphur give corrosive petroleum on treatment with acid. In the course of this work Di-n-heptyl disulphide, b.p. 164°/6 mm., $d\,\frac{15.5}{15.5}$ 0·9082 was prepared for the first time. The corrosive substance was finally identified as a polysulphide (almost certainly a trisulphide), and received final proof by the surprising observation that when isobutyl or isoamyl mercaptan in petroleum solution is shaken with powdered rock sulphur, or when sulphur dioxide followed by hydrogen sulphide is let into the solution, a polysulphide is produced.

Diethyl trisulphide $C_2H_5{\cdot}S{\cdot}S{\cdot}S{\cdot}C_2H_5$ b.p. 78°/5 mm., was synthesised from ethyl mercaptan and sulphur dichloride in the presence of pyridine or sodium ethoxide, and was found to possess all the characteristics of the corrosive substance under investigation. The concentrations of mercaptan in these experiments were much greater than those encountered in industrial practice, and it is not suggested that appreciable amounts of polysulphide is likely to be formed when commercial distillates are sweetened by means of doctor solution and elementary sulphur. H. E. T.

742. Composition of Paraffin Wax. S. W. Ferris, H. C. Cowles, Jr., and L. M. Henderson. *Ind. and Eng. Chem.*, 1929, 21, 1090.

The variations in the crystalline behaviour of the solid constituents of petroleum have given rise to three theories on the composition of these waxes: (1) the proto-pryo-paraffin theory of Laloziecki, which postulates the existence of an amorphous wax (probably branch-chain) which upon distillation cracks to the crystalline (straight-chain) variety; (2) theory of Gurwitsch, which attributes the differences in behaviour to the properties of the crystallising medium; and (3) the "soft wax" postulate of Buchler and Graves, which attributes the peculiarities to an "impurity" termed "soft wax." The present work was undertaken to investigate further the nature of the compounds present in paraffin wax.

Oil-free wax was recovered from intermediates by successive recrystallisations from ethyline dichloride at —18° C. These were fractionally distilled at an absolute pressure of less than 1 mm. H_g., using a 5 ft. column. The boiling ranges of the individual fractions were determined by vacuum assay distillations at 10 mm. pressure.

Fractions having identical 50 per cent. boiling points or refractive indices varied widely in melting points. From these fractions two waxes were prepared by mixing certain distillate fractions. Each of these mixes was fractionated as before and the middle cut selected for recrystallisation. The solvent used was ethylene dichloride. Each final fraction was recovered as a crystal at least eight times. It was assumed that if wax consisted of true paraffins contaminated with a "soft wax" impurity the recrystallisations would result in separation and purification of the two constituents. In the six different fractions finally secured no such tendency was found. Various physical properties of these fractions were determined.

The 50 per cent. boiling points are substantially the same, but what variation exists is in the opposite direction from that anticipated from the melting points, which vary from 59·9° C. to 29·4° C. Maximum difference in molecular weight is slightly less than two carbon atoms and the highest melting fractions exhibit the lowest molecular weight.

It is believed that the evidence presented is sufficient to warrant the conclusion that petroleum wax contains in addition to straight-chain, saturated hydrocarbons, a number of other types of hydrocarbons, which are probably branch-chain paraffins rather than unsaturated or cyclic compounds. W. L.

743. Catalysts for the Formation of Alcohols from Carbon Monoxide and Hydrogen. M. R. Fenske and P. K. Frolich. *Ind. Eng. Chem.*, 1929, 21, 11, 1052.

A study has been made of the formation of alcohols from carbon monoxide and hydrogen, by using ternary catalyst mixtures, consisting of the oxides of copper, zinc and chromium in the molar ratio 49 : 43 : 8. The catalyst possesses a considerably higher activity for both the decomposition and synthesis of methanol than any of the binary systems, copper-zinc and chromium-zinc discussed in previous papers. The ternary catalyst is active at low temperatures, gives a relatively high conversion and is particularly applicable for the synthesis of methanol. In the decomposition experiments it was found that there is a tendency for the catalyst to deteriorate with time, but its activity can be restored by oxidation followed by reduction.

The reduction of the catalyst with methanol vapour is apparently preceded by decomposition of the alcohol into carbon monoxide and hydrogen. The carbon monoxide appears to be the active reducing agent, while the hydrogen comes through unchanged. H. E. T.

744. Specific Heats of Mineral Oils. L. M. Henderson, S. W. Ferris and J. M. McIlvain. *Ind. and Eng. Chem.*, Analy. Ed., 1929, 1, 3.

This paper describes experiments conducted to determine the specific heats of various oils, both distillates and residues, from 0° C. to 250° C. A table of physical properties (density, flash point, viscosity and assay distillation) of the various oils is given, together with a summary of the experimental results. These are also shown in the form of a graph and are compared with results obtained by other workers. The apparatus consisted of a pot calorimeter fitted with a total immersion electric heater, paddle and sensitive mercury thermometer reading to ·04° C. A brass barrel of known heat capacity was capable of being fixed on the stirrer shaft below the surface of the oil. By the use of this it was possible to obtain two sets of conditions with the apparatus, the quantity of oil being different in both cases. Hence, by performing experiments with and without the barrel in position, the heat capacity of the volume of oil displaced by the barrel could easily be obtained by difference. Errors due to heat loss were eliminated in this way. The apparatus was reliable to within ± 1 per cent. The conclusion arrived at is that for engineering purposes the specific heat at high temperatures may be calculated by the equation due to Fortsch and Whitman :—

$$\text{Specific heat} = \frac{(\tau + 670)\,(2{\cdot}10 - \text{sp. gr. @ } 60^\circ \text{ F.})}{2030}$$

where τ = temperature in ° F. P. D.

745. Chemical Reactions in Gases by Means of Electrical Discharges. P. H. Hull and Imperial Chemical Industries Limited. E.P. 317,920. August 29, 1929. Appl. May 31, 1928.

In chemical reactions which are brought about by means of an electrical discharge, such as the conversion of methane into acetylene, the gases are passed in violently turbulent stream through a cylindrical or conical zone of discharge in a direction parallel to the discharge itself. The discharge may be between the rims of two coaxial tubes which may rotate or reciprocate relative to each other. A suitable apparatus consists of two aluminium tubes so arranged that the gas enters the apparatus by the inner tube and leaves by the annular space between the tubes. An arc is struck between a carbon electrode and the end of the tubes. The stream of gas tends to carry the arc into the annular space between the tubes. When the apparatus is used to treat hydrocarbons a slight deposit of carbon makes it necessary occasionally to adjust the distance of the carbon electrode. Sulphur-free coke oven gas containing 25 to 35 per cent. of methane, when passed through a high-tension arc maintained by 4000 volts 40 cycles A.C. gave a gas containing 3 to 5 per cent. acetylene when passed through the arc at a speed of 250 ft. to 300 ft. per second. T. A. S.

746. Manufacture and Production of Valuable Hydrocarbons. J. Y. Johnson, from I. G. Farbenindustrie Aktiengesellschaft. E.P. 316,422. Aug. 1, 1929. Appl., July 13, 1928.

In the production of valuable hydrocarbons (*e.g.*, butadiene) by heating to redness a mixture of acetylene and an olefine (ethylene), glass, quartz, porcelain, iron, nickel, copper, silver and many other materials cause extensive deposition of carbon. It is now found that this may be avoided by ensuring that the heated gases come into contact only with elements of the fourth group of the periodic system—*e.g.*, tin, silicon, lead, carbon (as graphite), or with zinc or aluminium or alloys containing substantial amounts of these

metals. These materials not only do not cause deposition of carbon, but have a catalytic action; they are therefore advantageously disposed on suitable supports (*e.g.*, plates). Moreover, anhydrous zinc, ferric or aluminium chlorides have a condensing action. The process may be carried out under elevated, atmospheric or diminished pressure. Usually pressures of about 50–100 atm., or even 200 atm., are employed. The reaction temperature is usually 200–800°, preferably 300–600°. The gaseous mixture employed may consist of equal parts of olefine and acetylene, or the former may be in excess. Such mixtures may be obtained by the pyrogenesis of tars, oils, bituminous coals, etc., and then contain 20–40 per cent. of olefines and 1–2 per cent. of acetylene. The gas mixture may be diluted with one or more extraneous gases—*e.g.*, nitrogen, methane or ethane, carbon dioxide or carbon monoxide. In the following examples the parts are by weight:—

A mixture of equal parts of ethylene and acetylene is passed vertically downwards through an aluminium tube, externally heated to 525–550°, containing metallic silicon disposed on a plate. About 39 per cent. of the reaction mixture is converted, and 89 per cent. of the material converted is transformed into an oil of b.p. up to about 200°. There is no formation of tar and no appreciable deposition of carbon.

When the same gas mixture is heated in a manganese-copper tube coated internally with zinc, 5·5 per cent. of the gas is converted into a liquid—of b.p. about 60–80°—consisting almost entirely of benzene. Only traces of butadiene and methane are present in the effluent gas. No deposit of carbon is found in the tube after 120 hours' use.

Benzene is also the chief product if the operation is performed similarly at about 600° in a brass tube.

An iron autoclave, coated internally with tin and then with powdered silicon, and in which silicon is disposed on plates, is charged to an initial pressure of about 50 atm. with a mixture of 20 per cent. of acetylene, 60 per cent. of ethylene and 20 per cent. of nitrogen. On being heated to 370–400° the reaction commences, and the same temperature is then maintained for about 6 hours. On cooling, 72 per cent. of the mixture of ethylene and acetylene is recovered as an oil, 90 per cent. of which boils up to 150°. The catalyst remains free from tar or carbon.

A mixture of 9 parts of ethylene and 1 part of acetylene is passed at 10 litres per hour through a manganese-copper tube coated internally with tin, 18 mm. in diameter, which is heated over a length of 60 cm. to about 600°. A continuous conversion of 4 per cent. of the gases is obtained, without any deposition of carbon. The condensate obtained on cooling to low temperatures contains 13 per cent. of butadiene. The remainder of the conversion product has b.p. up to about 95°. The residual gas contains ethylene, acetylene, a little propylene and butylene, and also 0·5 per cent. of methane. It can be recycled after adding acetylene, which is the chief constituent consumed.

W. S. N.

747. Manufacture and Production of Unsaturated Hydrocarbons. J. Y. Johnson, from I. G. Farbenindustrie Aktiengesellschaft. E.P. 315,895, July 16, 1929. Appl., March 16, 1928.

Mineral oil, tar, etc., is cracked at 600–800°, in the presence or absence of gases or vapours (*e.g.*, steam), and, if desired, in the presence of catalysts that facilitate cracking. Condensates are then separated from the resultant gases by fractional condensation, and from these condensates unsaturated hydrocarbons are recovered in a pure state by fractional distillation. Suitable pressures are 150–250 atm. Catalysts specially suitable are silicates either alone or in conjunction with difficultly reducible metallic oxides such as chromium or molybdenum oxides; the oxide may be carried on the silicate.

Other suitable catalysts are copper, iron, chromium, zinc, or their alloys. The resulting olefine-bearing gases contain 20–34 per cent. of ethylene, 4–12 per cent. of propylene, 1–5 per cent. of butylene and amylene, and the remainder consists of methane, hydrogen, carbon monoxide and nitrogen. A detailed example is given of the production of pure olefines from such a gas mixture.

W. S. N.

748. Recovery of Diolefines. J. Y. Johnson, Assr. to I. G. Farbenindustrie A.-G. E.P. 319,025. March 12, 1928.

Diolefines may be separated from mixtures with mono-olefines or saturated hydrocarbons by treating with certain organic solvents at suitable temperatures and pressures, the diolefines being thereby dissolved and separated from the mixture.

As suitable solvents are instanced ethylene chlorhydrin, glycol mono- or di-acetate, glycol monomethyl ether, glycerin di- or tri-acetate, lactic acid nitril, diethyl tartrate, and furfural aromatic bases such as aniline, toluidine or phenyl hydrazine can be employed.

It is suggested that the efficiency of the above solvents is connected with their dielectric constants, as furfural, whose dielectric constant is 41·7, is very much more effective than benzaldehyde of dielectric constant 18, and aniline and phenyl hydrazine with dielectric constants of 7·3 and 7·15 respectively have better solvent action for diolefines than has toluidine, whose dielectric constant is 6.

If a mixture of butylene and butadiene is passed into furfural at 0° C. almost complete separation can be effected by solution of the butadiene. Mixtures of easily condensable gases or low boiling point liquids such as pentane and isoprene can be readily separated in the liquid phase by the use of a suitable solvent such as ethylene chlorhydrin.

H. B. T.

749. Improvements in the Manufacture and Production of Unsaturated Hydrocarbons. J. Y. Johnson. E.P. 317,500. Aug. 14, 1929. Appl., April 14, 1928.

By means of this invention a process is provided for the manufacture and production of unsaturated hydrocarbons, and in particular of diolefines, by the catalytic dehydration of the corresponding hydroxylated hydrocarbons. The method consists in passing the initial material in the gaseous phase over catalysts consisting of oxygen salts of the acids of phosphorus, which have an acid reaction under the working conditions, sodium phosphate and aluminium phosphate being excluded. A small quantity of free acid or a substance which will produce free acid under the working conditions, is usually incorporated with the catalyst. The following example illustrates one method of carrying out the process :—

100 parts of secondary calcium phosphate are made up into a stiff paste together with 10 parts of secondary ammonium phosphate, 40 parts of water and 10 parts of graphite, which has been boiled out with hydrochloric acid and washed. This paste is then passed through an extrusion press. The catalyst in the form of granules, furnishes a yield of over 90 per cent. of butadiene from 1·3-butylene glycol, in an almost pure state.

Several examples are provided illustrating the manner in which 1·3-butylene glycol, ethyl alcohol, cyclohexanol, 2-methyl-1·3-butylene glycol, may be transformed into butadiene, ethylene, cyclohexane and isoprene respectively.

H. E. T.

750. Improvements in the Manufacture and Production of Organic Compounds from Oxides of Carbon and Hydrogen. J. Y. Johnson. E.P. 317,808. Aug. 19, 1929. Appl., May 18, 1928.

This invention relates to the production of organic compounds, and in particular those containing oxygen such as alcohols, acids, esters, ketones, fats and hydrocarbons. The process consists in treating gaseous mixtures, composed of oxides of carbon and an amount of hydrogen or a gas rich in hydrogen, at a pressure of twenty atmospheres, with a catalyst consisting of an element from the first sub-group of the Periodic System and a large amount of an element from the eighth group of the Periodic System, the catalyst being free from cadmium and thallium, if the gaseous mixture contains oxides of carbon in more than 25 per cent. excess over the hydrogen present. H. E. T.

751. Chemical Effects of Semi-Corona Discharge in Gaseous Hydrocarbons. S. C. Lind and G. Glockler. *J. Amer. Chem. Soc.*, 1929, **51**, 2811.

It is shown that, like ethane, the lower hydrocarbons, methane, propane, butane and ethylene condense to liquid and solid hydrocarbons in semi-corona discharge, obtained from a central aluminium rod in a pyrex glass cylinder. The condensation of saturate compounds was brought about by the elimination of hydrogen and some methane as under alpha radiation. In series gas flow through a number of discharge tubes, the amount of liquid condensate increased in successive tubes. If the rate of flow were so slow that depletion of reactant became considerable, then a maximum of liquid yield was attained in an intermediate tube. The liquid products obtained from different hydrocarbons were similar in physical properties when obtained at the same trap temperature, and very complex in nature. Solid hydrocarbons were obtained in small quantities in the Siemens tube and in larger quantity, equal to that of liquid, in the corona tubes. The solid was resinous and inert towards solvents and chemical reagents except strong oxidising agents. In corona discharge, a small amount of free carbon was deposited on the metal electrode in tree-like formation. A close correlation of index of refraction, molecular weight, density, colour and viscosity characterised all the liquid products. Increase in all these properties was inversely proportional to the yield, for which a simple explanation is given. W. H. T.

752. The Action of Sulphuric Acid on Olefines. W. R. Ormandy and E. C. Craven. *J.S.C.I.*, 1929, 48, **40**, 293 T.

In this paper the authors reply to a criticism of a previous paper on the action of sulphuric acid on olefines, made by J. F. Norris and H. S.Davies. The point on which the criticism was raised concerned the practical possibility of manufacturing higher alcohols from cracked spirit by washing with sulphuric acid, and then recovering the alcohol by dilution and subsequent distillation. H. E. T.

753. Heat of Fusion Determined for Many Normal Hydrocarbons in Oil. G. S. Parks and S. S. Todd. *Oil & Gas. J.*, 1929, 28, 19, 143.

The heat of fusion of a number of paraffin hydrocarbons has been determined by the above authors. The data quoted in this article were derived principally by calculation, following the determination of the heat of fusion of four pure normal hydrocarbons—namely, C_9H_{18}, $C_{20}H_{42}$, $C_{21}H_{44}$ and $C_{32}H_{66}$. The calculations were made by means of the formula :—

$$\Delta^{St} = 4n^{\cdot 888} - 1{\cdot}43$$

in which Δ^{St} = entropy units per mol.

n = number of carbon atoms in the chain.

This formula was derived from a curve obtained by plotting the molar entropy of fusion against the number of carbon atoms. H. E. T.

754. Synthesis and Free Energy of Methane. M. Randall and A Mohammad. *Ind. Eng. Chem.* **21**, **11**, 1048 (1929).

This paper presents new data upon the equilibrium between graphite, hydrogen and methane. The apparatus used in this work was similar to that employed by Randall and Gerard (*Ind. Eng. Chem.*, **20**, 1335 (1928), with the exception of the reaction furnace and the automatic sampler. The hydrogen was prepared electrolytically, while the methane was obtained by fractionation of natural gas. They were purified by freezing and subsequent boiling out of the low boiling-point gases (nitrogen, oxygen and carbon monoxide) under reduced pressure. All the gases were analysed by means of a standard Burrell gas analysis apparatus, the burette of which was maintained at a constant temperature by water which was continually circulated from a thermostat. The catalyst employed was reduced nickel with ceria as a promoter, the supporting material being granulated Nonpareil brick.

The equilibrium constant K was calculated as follows:—If P is the total pressure and $N(CH_4)$ and N (H_2) are the molar fractions of methane and hydrogen, then

$$K = N(CH_4)/P.N^2(H_2)$$

The average value of the free energy of formation of methane was found to be:

$\Delta F^\circ_{298\cdot1} = -11{\cdot}573$ cal., which is in good agreement with the value.

$\Delta F^\circ_{298\cdot1} = -11{\cdot}582$ cal., obtained previously by Randall and Gerard.

H. E. T.

Natural Gas.

755. Analyses of some Natural Gasoline Gases before and after Treatment. H. C. Allen. *Ind. Eng. Chem.* (Anal. Ed.) 1929. I., **4**, 226.

In this article the data on three natural gasoline plants, using mineral seal absorption oil are recorded. Combustion analyses of the gases are given for the sake of comparison but are found to be quite different from the actual analyses. The methods of analysis employed followed in a general way the published methods, but numerous modifications were followed. The gases were fractionated by means of liquid air at different temperatures, the fractions obtained being analysed by combustion methods. To eliminate variations in molecular volumes and any absorption of carbon dioxide after combustion, the higher fractions were calculated, from the combustion data by combining the carbon dioxide and contraction and using this combined value with the oxygen consumed. Three or four fractionations gave a rather poor product, but in practically all cases the combustion data indicated the presence of two hydrocarbons, while for some reason the propane fraction appeared to be the easiest to obtain in a fine condition. H. E. T.

756. Method of Heating Natural Gas Lines. L. G. E. Bignell. *Oil & Gas J.*, 1929, **28** (20), 191,305.

A problem causing trouble to the natural-gas engineer is the freezing of valves and fittings by the expansion of the gas. It has been the practice to run duplicate lines from the well-head to ensure at least one line in service. In the new system no duplicate lines are needed, one 2-in. high-pressure line running from the well with one gate valve set about 12 ft. in front of the heater.

The heater consists of about 12 ft. of 16-in. casing. A leader is welded to each end and the 2-in. pipe passes through as in a return-flue boiler, having an effective length of about 36 ft. An opening on the top side permits expansion and introduction of water. One inch of crude oil on the surface

of the water reduces evaporation. The water is heated to about 150° F. by a gas heater on the under side and maintained at this temperature by a thermostatic control. When no gas is passing the heat travels back to the control valve and keeps it free from air.

The cost of this heater is about 50 per cent. less than the old system and operating expenses about one-third. With a system handling 3 to 10 million cu. ft. of gas, the heating gas amounts to 2,400 to 4,800 cu. ft. per day. G. S.

757. GASOLINE PLANT GATHERING SYSTEMS. J. C. Bolinger. *Oil & Gas J.*, 1929, **27** (47) 150.

In the design of pipe lines for the transmission of gas it is necessary to make use of some formula expressing the relations to each other of the quantity, initial and final pressures, diameter and length of line. Many such formulæ have been proposed. In nearly all these formulæ the flow is stated as varying as the square root of the fifth power of the pipe diameter and either the coefficient of friction is considered constant or a different coefficient is given for each size of pipe. These formulæ are awkward to use when it is desired to ascertain the diameter of the line. Weymouth gives a formula based on isothermal flow and the variation in the value of the coefficient of friction is provided for by changing the power of the diameter without complicating the formula.

Weymouth's formula in the form most generally used is :—

$$Qs = \frac{15385 \times \sqrt{P_1^2 - P_2^2} \times \sqrt{d^{5.333}}}{\sqrt{G.T.L.}}$$

Where :—

Qs = Quantity of gas per 24 hrs. at 60° F. and atmos. press.
P_1 = Initial pressure in lbs. per sq. in abs.
P_2 = Terminal pressure in lbs. per sq. in abs.
C_1 = Specific gravity (air = 1).
T = Flowing temperature (F ° abs.)
L = Length of line in miles.
d = Inside diameter of line in inches.

This becomes :—

$$Qs = \frac{871 \times \sqrt{P_1^2 - P_2^2} \times \sqrt{d^{5.333}} \times M}{\sqrt{L}}$$

where M = A multiplier for conditions of gas other than 60° F. and 0·6 gravity (Air = 1)

$$M = \sqrt{\frac{0{\cdot}6 \times 520}{G \times T}}$$

The formula shows that the quantity will vary inversely as the square root of the length but the variations of pressure drop with length and quantity are not such simple functions. For a constant Qs, d, T & C_1 the formula becomes:

$$\sqrt{L} = \sqrt{P_1^2 - P_2^2} \times K.$$

From this the pressure drop varies as L divided by the sum of the initial and terminal pressures times the constant squared. Considering the variation of quantity and pressure drop we have a constant L, T, d & G, so :—

$$Qs = \sqrt{P_1^2 - P_2^2} \times K$$

Thus the pressure drop varies as the square of the quantity divided by the sum of the initial and terminal pressures times the square of the constant.

Graphical solution of the formula is explained and the necessary curves are given. The use of these curves is explained by a practical example.

The article concludes with a description of the layout of a more or less simple gathering system with a brief explanation of some of the conditions that must be considered. W. L.

758. Treatment of Casing-head Gasoline. E. C. Herthel, assig. to Sinclair Refining Co. U.S.P. 1,733,800, Oct. 29th, 1929. Appl. June 13th, 1927.

Gas or vapour mixture, while substantially free from water or water vapour, is subjected to treatment with solid cupric chloride and the so treated gas or vapour mixture subsequently subjected to a compression or absorption treatment to obtain the more readily condensable vapours in a liquid form. During the cupric chloride treatment the "sour" elements present in the gas are converted into an inactive form but part of the reaction products may remain in the gas and vapour mixture and appear in the liquid condensate obtained from it. These reaction products appear to be negative to the sodium plumbite test. The reaction products appearing in the liquid condensate may be effectively removed by treatment of the condensate with solid absorbent material such as Fuller's earth or similar absorbent earth's or clays. W. L.

759. Purification of Gas Containing Sulphuretted Hydrogen. J. Y. Johnson, from I. G. Farbenind. A.-G. E.P. 319,396. Sept. 20, 1929. Appl. June 20, 1928.

In the process for purifying coal gas, producer gas, natural gas, etc., from H_2S by passing the gas with a suitable volume of air or oxygen over a catalyst composed of nickel, iron, cobalt, etc., or their compounds, with or without an oxygen carrier such as chromium, vanadium, silicon, carbon, or molybdenum, the temperature range of the reaction for quantitative oxidation of H_2S to sulphur oxides is narrow (300–350° C.). It has been found that this temperature range can be widened by the addition of up to 10 per cent., preferably 5 per cent., of lead, bismuth, copper, or their compounds, to the catalyst. Small additions of alkali metal or alkaline earth metal compounds have a similar activating effect. The catalyst metals are deposited on a carrier; *e.g.*, pumice stone, burnt fireclay, or silica gel, which is impregnated with a solution of the nitrates, thereafter being calcined. The temperature range is 100–700° C., preferably 200–400° C. The pressure may range from atmospheric up to 1000 atmospheres.

When the gases are contaminated with tar or oils carbon is deposited on the surface of the catalyst, but the efficiency may be maintained by allowing the initial temperature to rise by about 5° C. every three days.

The SO_3 or SO_2 to which the H_2S is oxidised may be recovered as ammonium sulphite-bisulphite liquor, in which case the ammonia content of the gas prior to scrubbing must be adjusted if necessary; the liquor from the first scrubber should be sufficiently acid for direct conversion to sulphate by heating under pressure; final traces of SO_2 are removed in subsequent scrubbers. L. H. A.

760. Art of Obtaining Gasoline Hydrocarbons. W. K. Lewis. U.S.P. 1,730,152. Oct. 1, 1929. Appl. Feb. 3, 1923.

A process and apparatus are described in which a gas containing gasoline hydrocarbons (*e.g.*, natural gas) is stripped of its gasoline content by passage up a tower countercurrent to a suitable absorption liquid (*i.e.*, a liquid which is non-volatile under the existing conditions and miscible with the hydrocarbons). The gas is introduced at an intermediate point of a gas and liquid contact zone and the absorption liquid is introduced above the gas inlet, being at least in part adjacent to the top of the contact zone. The latter is

heated near the bottom by means of a coil through which passes the hot stripped absorption oil, whilst it is cooled at the top by means of appropriate cooling coils.

W. S. C.

761. The Compression of Refinery and Casing-head Gases. W. J. Murray. *Ind. Eng. Chem.*, 1929, **21**, (10), 917–919.

Starting from the laws of Avogadro, Dalton and Raoult, the paper develops and illustrates a means of calculating the composition and quantity of both the liquid and gaseous phases produced by the compression of gases of known chemical composition.

The following fundamental equation is readily obtained for 100 lb. mols. of compressed material :—

$$\left(\frac{A_L}{L}\right) = \frac{A}{100\frac{P_A}{P} + L\left(1 - \frac{P_A}{P}\right)}$$

where the known quantities are :—

A = lb. mols. of component A present in 100lb. mols. of gas.
P = absolute pressure of system in lbs./sq. in.
P_A = vapour pressure of component A in the pure state under the given temperature conditions.

And the unknown quantities are :—

A_L = lb. mols. of component A in the liquid from 100lb. mols. of gas.
L = lb. mols. of total liquid produced.

This equation may be extended to any number of light homologous hydrocarbon constituents, A, B, C, D, etc., when

$$\frac{A_L}{L} + \frac{B_L}{L} + \frac{C_L}{L} + \ldots\ldots\ldots\ldots\ldots = 1$$

The complete set of equations may be solved simultaneously, but in practice it is more convenient to determine L by trial and error. The first step towards solution consists in putting $L=0$ in the *right-hand side* of the first equation. If this gives $\frac{A_L}{L} > 1$, liquefaction will most certainly take place. If $\frac{A_L}{L} < 1$ then $\frac{B_L}{L}$, $\frac{C_L}{L}$, etc., must be determined. If the sum of these terms > 1 liquefaction will take place. At the dew point the sum $= 1$. If the sum < 1 no liquefaction will occur under the given conditions.

Assuming liquefaction does take place, suitable values for L should be substituted in the right-hand side of the equation so that the sum $\frac{A_L}{L} + \frac{B_L}{L} + \frac{C_L}{L} + \ldots\ldots\ldots$ is nearly 1. The correct value of L may then be determined by interpolation, and A_L, B_L, C_L, etc. may be determined, also the lb. mols. of A, B, C, etc. in the residual gas.

If steam is present in the gas the vapour pressure of pure water at the given temperature must be subtracted from the total pressure, giving a modified P. From the final gas volume and the partial vapour pressure in it the weight of water in the gas phase may be calculated.

If air is present the assumption must be made that air is insoluble in gasoline : the mols. of air in the liquid are assumed to be zero.

For purposes of heat calculations the value of n for the compression curve may be taken as 1·19 for a rich gas.

A practical chart is reproduced for rapid determinations of the volume of 1lb. mol. of gas at various temperatures and pressures. L. H. A.

762. GASOLINE CONDENSER, STABILISER, FRACTIONATOR AND DEHYDRATOR. H. R. Otto. U.S.P. 1,728,440, Sept. 17, 1929. Appl., June 20, 1927.

The invention relates to an apparatus designed to remove undesirable highly volatile constituents from casing-head gasoline, produced, for example, by absorption of wet gas in mineral seal oil. The apparatus consists of a ve ical cylindrical shell having a bottom chamber, a central "reaction" zo ıe, and a top chamber. The reaction zone is built up of a series of superimposed cylindrical sections concentric with the main shell, but of somewhat smaller diameter. The base plate of each section is of such diameter as to fit nicely with the walls of the outer shell, and the joint is rendered substantially fluid tight with packing. Each cylindrical section is divided into two chambers by a central horizontal plate. These plates and those areas of the base plates falling within the reaction zone are perforated with small holes, the size of which is made to decrease progressively from the bottom plate upwards. The topmost plate and each base plate carries a central overflow funnel and tube discharging into a seal pot on the middle plate of the next lower section. Near the base of the upper and lower chambers of each section a series of ports is drilled communicating with the annular space, the upper series of ports being staggered relative to the lower series.

The apparatus operates as follows. Wild gasoline vapours from the stripping still at about 200° F. and 20–30 lb./sq. in. are led into the bottom chamber, and travel upward through the perforated plates of the reaction zone, countercurrent to cold stabilised or blending gasoline which is fed on to the topmost plate. The gasoline, unable to percolate through the perforations on account of the rising vapours, flows alternately inwards and outwards on the top and middle plates respectively of each section, downward transference being effected alternately by central overflow tube or outer annular chamber. The bottom-most plate carries an overflow pipe with seal pot attached, which conveys the stabilised gasoline into the bottom chamber.

The repeated intimate contact between the gasoline and the vapours results in the solution of all the condensable hydrocarbon vapours. the uncondensable vapours being drawn off from the top chamber. The stabilised gasoline is drawn off from the bottom reservoir, and water is removed periodically from the bottom. After the initial supply of scrubbing gasoline, some of the stabilised make may be pumped back on to the top plate through a cooler. L. H. A.

763. FLOW-METERS FOR NATURAL GAS. *Petr. Times*, 1929, **22**, 463.

Meters for the measurement of gas have proved quite reliable as regards freedom from breakdown, but have frequently given dissatisfaction due to their design and to the method of measurement. Apart from mechanical difficulties, the main one is the correct reading of the scale on the diagram, which usually has logarithmic divisions. The method of calculation is to determine the average radius and deduct from this the constant representing the distance from the centre to the zero line, thus obtaining the average differential pressure. The same procedure is adopted in order to obtain the average static pressure, and the two average pressures are multiplied

together. An alternative method to obtain an average value would be to multiply the individual differential pressures by the static pressure prevailing at a given time. The general formula used in practice, however, leads to a discrepancy in the order of 10 per cent.

A newer type of meter has been developed which contains no fluid, its only moving part being a metal diaphragm connected to a needle value. The inertia of the diaphragm and needle is so small that it responds instantly to any sudden change in volume of flow. The meter is based on the "deviated flow" principle, wherein the main flow is tapped and a partial flow diverted which remains proportional in weight to the main flow. This deviated flow is allowed to expand and is measured in an apparatus similar to an experimental gas meter at atmospheric temperature and pressure. Due to the provision of the diaphragm and needle value, a pulsating flow of gas can be measured; for the diaphragm adjusts itself immediately to any alteration in flow, and the proportion represented by the by-passed flow consequently remains unaltered. The connections between the main piping and the flow deviator are so placed that they run downwards and are provided with downward extensions for the collection of condensate, dirt, etc. For greater security, a cloth filter is inserted in order to prevent any fine dust from reaching the flow deviator. This type of meter can be read with ease, there is no need to resort to any process of planimetering, and all that is necessary is to read the flow meter at a definite hour each day. The difference between two readings is taken and multiplied by the appropriate constants. Fluctuations in the load are recorded on a continuous strip of paper, and describe a curve which is a direct linear chart of the load. W. H. T.

764. United States Helium Industry. *Petr. Times*, xxii., 558, 564.

The discovery, in Colorado, of a natural gas containing over 7 per cent. of helium has recently been announced. It is considered that this discovery will greatly reduce the cost of production of helium, as hitherto the highest percentage of helium found in natural gas was 3·6 per cent. in a Utah occurrence, whilst the state gas field of Petrolia contains not more than 1·75 per cent.

As is generally known, helium is utilised for the inflation of airships, as it has the great advantage in being non-inflammable. The cost of production is its greatest disadvantage. Originally it is said to have cost 2,000 dollars per cu. ft., but development of commercial processes had reduced the cost to approximately 4–6 cents per cu. ft.

The Petrolia field was selected by the U.S. Government as the source of helium on account of the prospects of abundant supplies of gas over a long period. It is stated that the field produces 60,000,000 cu. ft. of natural gas per month. The gas is unique, as it is of a relatively low heating value, due to its nitrogen content, but it contains a high gasoline content for what is commonly called a dry gas.

The basic principle of the processes used for the recovery of helium is the liquefaction of the constituents, one after another, until finally only helium is left in the gaseous condition. R. P.

765. Ends Sought in Applying Gas Lift. R. H. Pence. *Oil and Gas J.*, 1929, 28 (14), 106-107.

In examining a well being considered for an installation of the lift necessary information is (1) depth of well and producing formation, (2) nature of producing formations, (3) size of hole and controlling size of casing or oil string, (4) mechanical condition of the well, (5) initial production, and whether

well flowed naturally or not, (6) rate of decline of oil and gas, and gas factor curve, (7) nature of oil production from formation, *i.e.*, whether water or gas drive, or both, and relation of each to oil produced, (8) whether bottom water present, (9) present rate of production compared with nearby wells in the same pool, (10) present method of production. Further specific information relating to the operating characteristics of the well that is necessary is (1) gravity of the oil, (2) gasoline content of the oil, (3) gasoline content of the gas, (4) gravity of the gas, (5) well temperature, (6) solubility of the gas in the oil at varying temperatures and pressures, (7) formation or rock pressure of the well (several methods for determining same described), (8) rate of production under different differential pressures (methods described). Various devices for determining the depths of liquids and the pressure resulting from corresponding heads, and a depth bomb, are described.

H. S. G.

766. Artificial Methods of Oil Recovery. C. H. Pishny. *Oil & Gas J.* 1929, 28 (14), 41, 172.

The article deals briefly with the effects of the so-called artificial methods of production on the ultimate recovery of oil. The methods discussed are the air-gas left, controlled pressure operation, repressuring by air or gas, or both, and artificial water drive.

It is shown that the air-gas lift may not increase ultimate production, but that its success depends largely on general local conditions. Controlled pressure is a gas conservation measure and will increase ultimate production in varying degrees. Repressuring whether carried out early or late in the life of the well or property will in most cases be successful in greatly increasing the ultimate yield. Water flooding, the most efficient agent for removing oil from depleted sands, will greatly increase the ultimate production, but at such a high cost as to be limited to oils having a high market value.

W. H. S.

767. Steam Economies at Gasoline Plants. W. L. Rifenberick. *Oil & Gas J.*, 1929, 28 (17), 46, 159-161.

In the natural gasoline refining industry economies in large power plants are being made through the use of higher pressures and steam temperatures, continuous boiler ratings of 300 per cent. and 400 per cent. being possible with less maintenance and repairs. Boilers have been equipped with water walls to minimise refractory wall repairs, feed water economizers, steaming economizers and air heaters added after economizers, whilst superheaters are installed between banks of boiler tubes or occupy the radiant section of the combustion wall space. Where oil fuel is used in the mechanical type of burner, furnace temperatures may reach 3050° F., whereas using steam atomizers under the same conditions2, 600° F. is rarely exceeded. The use of mechanical burners in conjunction with water walls gives greatly increased efficiency. With feed water economizers utilising waste gases from a boiler the boiler heating surface may be reduced 20 per cent., and while the final cost will be about the same for a complete boiler unit, a net gain will be reflected in greater boiler ratings and maximum efficiencies. Modern boiler, practice is to proportion the boiler heating surface, economizers and air heaters, so that the total overheat cost of steam generation is less than the usual practice of three pass boiler with efficiency averaging 75 per cent. Detailed comparisons are made between duplex, reciprocating and centrifugal pumps, and it is shown that while the first cost of the turbine-driven centrifugal pump in small sizes is higher than the first cost of a reciprocating pump, the operating and maintenance charges are much less. Turbine units are reviewed and

finally the following plan, for the use of steam in gasoline plants is proposed. Generate steam at 200 lb. pressure and 200° F. superheat. Exhaust steam from a turbine will give a superheated steam above the temperature of the oil in the evaporator, *e.g.*, a 150-h.p. turbine will exhaust steam against 30 lbs. back pressure at 429° F. Any surplus steam can be condensed to reclaim water for the boiler plant. The balance of superheated steam from the boiler plant is put through heating coils in the evaporator to be desuperheated before being put in the preheater. The steam for the boiler feed pumps should be taken from the preheater condensate tank to remove air and increase the heating efficiency of the preheater. The condensate from the preheater is pumped back into the boiler without reduction of pressure in order to save the temperature of the hot water and prevent evaporation. The water from the primary condenser will be 85–90° F. of the steam admitted to the evaporator, providing the outlet temperature of the vapours from the primary condenser is approximately 150° F. The balance of the water from the secondary condenser will be 10—15 per cent., and will have a temperature of about 75° F. Both will go to the hot well with a temperature about 140° F. Any gasoline carried through the water will escape at this point, and the cold make-up water will be admitted to the hot well with a liquid level control. The boiler feed pump will exhaust to a feed water heater to heat the boiler feed water to as high a temperature as possible, and by this means the boiler plant is operated with very soft water, and a further advantage is the elimination of lubricating oil from the condensate. H. S. G.

768. Analysis in Oil Absorption Operation. W. W. Robinson, Jr. *Oil & Gas J.*, 1929, **28** (3), 54, 182.

When dealing with theoretical considerations of the phenomena of absorption, Dalton's and Raoult's laws come into considerable prominence. Starting with these laws as a basis the following equation is evolved :—

$$Ro = \frac{m_o}{G_o}\left(\frac{E_n\ p_n}{3.153\ P} - \frac{A\ G_a}{m_a}\right)$$

where R_o = Gallons of lean oil circulated per 1,000 cubic ft. of wet gas.
m_o = Molecular weight of lean oil.
G_o = Sp. gr. of lean oil.
E_n = Efficiency of absorption for a given fraction (n).
p_n = Vapour pressure of component n.
P = Total absolute pressure of vapour of which n is a part.
A = Total absorption expressed in G.P.M.
G_a = Sp. gr. of total absorption.
M_a = Molecular weight of total absorption.

Four examples of the application of this formula are given, three being of a practical nature and the fourth theoretical.

The treatment of the subject in this article is based on a condition of equilibrium at the bottom of the absorber tower. Under ideal conditions A=o at the top, the degree of equilibrium being determined by the fundamental analysis of the dry gas, for the constituent in question.

The formulæ presented act fairly well for oil rates up to about 30 : 1. Higher rates seem to distort things. This may be due to an upsetting of the equilibrium within the tower. W. L.

767. Natural Gas and Cracking Gas and Their Industrial Application. H. M. Stanley. *J. Inst. Pet. Techn.*, 1929, **15**, 516-552.

The composition of natural gas, some analyses of which are given, is first dealt with by the author who divides the methods of analysis into (a) those

in which the gas is liquefied with liquid air and the condensate subsequently fractionated at low temperatures and (*b*) those in which the gas is subjected to fractional condensation at low pressure in various temperature stages. The method of Burrell, Sibert and Robertson (*U.S. Bureau of Mines Tech. Paper* 104) falls into the former category, but is not suitable for plant control, while that of Shepherd and Porter (*Ind. Eng. Chem.* 1923, 15, 143) consists of modifications and improvements of this method. The apparatus devised by Podbielniak (*Oil & Gas J.*, Oct. 18, 1928) consists of an efficient fractionating column and still adapted to precise manipulation on small amounts of sample and operating at low temperatures. A similar fractionating column has been described by Davis (*Ind. Eng. Chem. Anal Edn.* 1929, 1, 61). In the latter category is included the method of Tropsch and Dittrich (*Brenn. Chem.*, 1928, 6, 169) in which the method is simple and the apparatus employed easily manipulated.

Commercially, the removal of gasoline from natural gas is effected by (*a*) compression; (*b*) absorption by oil; and (*c*) adsoprtion by materials such as activated charcoal. The stripped gas may be used for fuel, for the manufacture of carbon black or for thermal decomposition processes. Liquefied natural gas can be used as a fuel, a carburetting agent, a substitute for welding gas, a commercial solvent, a refrigerant or as a chemical raw material. High pressure gas from compression plants has been used for the repressuring of oil sands. Thermal decomposition of methane, etc., results in the production of carbon and hydrogen, the latter being suitable for Berginisation processes. Catalytic decomposition with steam results in the production of hydrogen suitable for the manufacture of synthetic ammonia. Chlorination provides a suitable base for the preparation of alcohols, while partial oxidation to formaldehyde and methanol can be carried out commercially. The pyrolysis of natural gas will result, according to the method employed, in the production of olefines or aromatics. The former may readily be converted into alcohols, glycols, etc. The pyrolysis of methane presents a separate problem, but under certain definite conditions, higher hydrobarbons can be prepared (Fischer. *Brenn. Chem.*, 1928, 9, 309). The disintegration of methane or its homologues by means of various types of electric discharge at atmospheric pressure results almost invariably in the production of acetylene, hydrogen, and traces of ethylene and higher liquid and solid hydrocarbons. This acetylene can be used for the synthesis of acetaldehyde, ethyl alcohol and acetic acid and can readily be polymerised to benzene and its homologues. Electrical disintegration of acetylene has been proposed as a method of producing carbon black.

The gas produced by cracking operations is produced by the liquid phase and vapour phase processes, but the composition of the gas depends on the type of plant, operating conditions and type of charging stock. Vapour phase cracking results in a greater yield of gas more unsaturated in character than the liquid phase. Many of the methods of analysis already given are applicable to these gases, while that of Manning Sinnatt and King (*Dept. Scien. & Indust. Res. Tech. Pap.* 19, 1928) consists in converting all the olefines into their bromine addition compounds followed by the fractionation of the resulting liquid under reduced pressure. Mixed olefines are liberated from the fractions by means of zinc-copper couple and are subsequently examined. The uses of cracking gas are very similar to those of natural gas with the addition that ethylene can be used as an anæsthetic, for the artificial ripening of fruit and for the formation of ethylene dichloride, alkyl chlorides, chlorhydrins, alcohols and ethylene glycol as bases for chemical manufacture. Controlled polymerisation has been suggested as a means of producing motor fuel. Further suggested uses for olefins are, the manufacture of diethyl sul-

phate from ethylene, oxidation of olefines to formaldehyde, and the utilisation of olefine gases for the manufacture of cyanides by interaction with nitrous oxide at high temperature.

W. H. T.

770. Some Uses of Oil and Gas Separators. C. L. Shirley. *Oil and Gas J.* 1929, **28** (16), 37, 147.

The physical-chemical laws under which a liquid mixture of various hydrocarbon moleculars of different characteristics may lose its single homogeneous equilibrium and come to rest under normal conditions as a multiplicity of stable homogeneous mixtures are complex, and therefore a perfect trap or separator cannot be designed as too many variables exist both in the operating conditions and in the oil itself.

Most separators used in high gravity fields use the separating principle of centrifugal action.

The gas throughput and oil throughput can be readily calculated for any separating system. It is a function of the five-halves power of the average minor axis of the smallest cross sectional area in the system. The velocity of the gas must be below the critical point at which speed it will carry entrained oil.

In practice, the capacity of a separating unit depends upon the volume that may be passed through it while the system functions within the critical limits of its component parts.

A general flow formula which has given good results is :—

$$Q = \frac{C\sqrt{[(P_1)^2-(P_2)^2]\,D^5}}{LG}$$

Where Q = Quantity in cub.ft. flowing per hr. at atmospheric pressure.

P_1 = Initial pressure in pounds per sq. inch absolute.

P_2 = Final pressure in pounds per sq. inch absolute.

D = Inside diameter of the pipe in inches.

L = Length of pipe line in feet.

G = Specific gravity of the gas.

C = A coefficient depending on the formula used.

In the examples given C=2782, and is derived from Towl formula.

For rapid calculations it has been found that this formula will approximate volumes within the accuracy of manometer readings. Three drawings are given, showing connections, as a well is being drilled in, when it becomes necessary to boost a natural flowing well and in the final stages of artificial flowing life. The original installation should be designed so that during the entire period of usefulness of a separator no connections to or from it need be changed.

W. L.

771. Flow Resistance of Gas-Oil Mixtures. L. C. Uren, P. P. Gregory, R. A. Hancock and G. V. Feskov. *Oil and Gas J.*, 1929, **28** (20), 148, 152.

Little attention has been paid to the resistance to flow of gas-oil mixtures rising in wells operated by gas-lift or by natural flow. At the lower end of the eduction tube the volume ratio of gas to oil is at a minimum, increasing to the maximum at the surface. The velocity of flow increases similarly, and the flow is undoubtedly turbulent.

The flow resistance is made up of two variables: (*a*) friction against the tube, and (*b*) internal friction in the fluid mass. The Fanning formula would comprise all the variables involved, which are related as follows:—

$$P = \frac{0{\cdot}323\ f\ LSV^2}{Z}$$

where P = pressure loss in lbs./sq. in.
S = sp. gr. of fluid.
D = internal diam. of pipe in inches.
L = length of pipe in feet.
V = velocity of flow in ft./sec.

$$f = \text{friction factor} = \frac{DVS}{Z}.$$

If *f* is made equal to $0{\cdot}00207 \times \frac{Z}{DVS}$, the Fanning formula becomes equal to the Poiseuille formula, in which

$$P = \frac{0{\cdot}000668\ ZLV}{D^2}$$

These expressions have been used in a series of tests at the University of California, and the following conclusions formed:—

1. Density and volume of gas-oil mixtures vary as logarithmic functions of the gas-oil ratio.

2. Increase in the volume ratio of gas to oil, at a given pressure, results in increased pressure loss per linear unit.

3. Increase in pressure for a given gas-oil ratio or a given flow velocity results in decreased unit pressure loss.

4. For any set of conditions, a critical gas-oil ratio probably exists at which pressure loss is at a minimum.

5. The friction factor, *f*, is a logarithmic function of $\frac{DVS}{Z}$.

The experimental work is being continued. G. S.

772. Unusual Features in Gasoline Plant. H. N. Wade. *Oil and Gas J.*, 1929, **28**, 19, 50, 186 and 189.

The world's largest absorption gasoline plant, near Monroe, La., is designed to handle 100,000,000 c. ft. per day of gas containing about 0·3 gallons better-grade gasoline per 1000 c. ft., at pressures of 300–600 lbs./sq. in. Six hammer welded oil absorption towers are provided, each 4 ft. diam. and 30 ft. high. The high pressure causes a large amount of fixed gas to dissolve in the oil, and weathering is effected in two stages. Saturated oil from the absorbers enters a high-pressure tank held at 150–240 lbs./sq. in., and then enters a low-pressure tank held at about 30 lbs./sq. in.; this makes for economy in the recompression of the weathered gas, which is added to the stripped gas and delivered at full pressure to the distributing pipe lines. The low-pressure cylinder of the compressor receives vapours from the low-pressure weathering tank; the compressed gas is cooled and delivered to the high pressure weathering tank, any gasoline condensing being returned to the low-pressure tank. The high-pressure cylinder receives gas from the high-pressure weathering tank and delivers the gas either upstream or downstream of the absorbing towers.

Fat oil from the low-pressure weathering tank is pumped through a heat exchanger and steam preheater to a vertical steam still operating at 30 lbs./

sq. in. pressure and using exhaust steam. Hot absorption oil leaving the still passes through the heat exchanger, a low-pressure lean oil pump, open type cooling sections in the water-cooling tower, a high-pressure pump, and then to the towers. Gasoline vapours and steam leaving the still pass to the dephlegmator, which consists of 24 passes of 2in. pipe arranged to lie in one horizontal plane in a shallow wooden trough on top of the cooling tower. The pipes are partly submerged in water and a constant vapour outlet temperature is maintained by thermostatic control of the water supply. Gasoline vapour, reflux oil, and water leave the dephlegmator and are separated; the gasoline vapour is condensed, passed to a gasoline dewatering tank having automatic float control of the gasoline and water outflow, and the uncondensed vapours are returned to the low-pressure weathering tank.

The absorption gasoline is stored under a pressure of 10 lbs./sq. in.; vapours liberated in the tanks are pumped into the low-pressure weathering tank. Vapours produced from the pressure storage tanks at the loading rack, and those liberated during the loading of tank cars are compressed to 100 lbs./sq.in. and cooled, when practically the whole of the vapours are recondensed. In this way, under summer conditions, about 300 gallons of gasoline are saved for each car loaded. L. H. A.

Motor Spirit.

773. A Comparison between American, Roumanian and Russian Petrols. W. Botte. *Rev. Petrolifère*, 1929, **340**, 1395.

A comparison is drawn between petrols derived from petroleums of American, Roumanian and Russian origin. The various hydrocarbons found in these petrols are described, and tables are provided indicating their distillation characteristics. From these characteristics the following conclusions are drawn:—

(1) Pennsylvanian petrols are very rich in saturated hydrocarbons.

(2) American petrols contain a large proportion of naphthenes and a small proportion of aromatics.

(3) Cracked spirits are rich in ethylenic and aromatic hydrocarbons, this being excellent from the point of view of anti-detonating properties.

(4) Russian petrols contain hydrocarbons belonging to the cyclopentane and cyclohexane series in large quantities, considerable quantities of aromatic and smaller quantities of ethylenic hydrocarbons, so that their employment in high compression engines is very possible.

(5) Java and Borneo spirits are extremely rich in aromatic hydrocarbons, so that these petrols are being utilised for the manufacture of explosives.

The paper concludes with a discussion on the relative volatilities of these hydrocarbons. H. E. T.

774. Improvements in the Manufacture of Low-boiling Hydrocarbons. I.G. Farbenindustrie Akt. E.P. 318,270. Aug. 27, 1929. Appl. Apr. 27, 1928.

The invention relates to the production of low-boiling liquid and gaseous hydrocarbons, in particular olefins and diolefins such as butadiene, from solid or liquid fuels or tarry products.

The process consists of subjecting the vapours or finely-divided substance to the action of an electrical discharge such as electric arcs or high frequency discharges of high temperature, for a short time, it being essential to remove the olefins and diolefins as rapidly as possible from the reaction zone.

For example, petroleum is distilled, and a flaming arc is produced between carbon electrodes, in the vapour space. The vapours pass rapidly through the electric arc so that the temperature of the reaction products does not rise above 700° to 800° C. One part of petroleum (boiling above 200° C.) furnishes 400 parts by volume of a gas having the following analysis :—$C_n H_m$, 22·8 per cent. ; CO, 0·6 per cent. ; H_2, 59·8 per cent. ; C_nH_{2n+2}, 16·2 per cent. ; N_2, 0·6 per cent. The C_nH_m hydrocarbons have the following composition :—C_2H_4, 26·1 per cent. ; C_3H_6, 20·7 per cent. ; C_4H_8, 3·5 per cent. ; C_3H_8, 30·8 per cent. ; and butadienes, 18·9 per cent. The gas also contains readily condensable hydrocarbons which are separated and returned for further conversion. The electrodes used in the process may be of any desired material, such as copper, copper alloys, chromium nickel alloys, iron, or carbon. R. P.

775. Conversion of Hydrocarbons of High Boiling Point into Others of Low Boiling Point. J. Y. Johnson, Assr. to I.G. Farbenind. Akt. E.P. 316,637. July 26, 1929. Appl. April 26, 1928.

The patent claims a process in which the initial high-boiling materials are treated at an elevated temperature and if desired under pressure, and in association with inert gases, in the presence of a catalyst consisting of or containing a salt of meta-phosphoric acid. W. H. T.

776. Desulphurising Hydrocarbon Oils. R. C. Moran. U.S.P. 1,692,756 Nov. 20, 1928. Appl. Jan. 18, 1927.

This invention relates to a process for the removal or reduction of the sulphur content of hydrocarbon oils, particularly petroleum, shale oil and other mineral oils, and more particularly the products derived from them. The oil to be treated is subjected to the action of metallic mercury while simultaneously subjecting the metallic mercury to a water solution of an alkali sulphide under atmospheric conditions. H. E. T.

777. Research on Motor Fuel. Sixth Report of the joint Benzol Research Committee of the National Benzol Association and University of Leeds. 1929.

It has been shown in Report 5, 1928, that the tendency of benzol to gum during storage or on oxidation could be diminished by using tri-cresol as an inhibitor. This material consists of a freshly distilled phenolic fraction boiling over a range of 195°–203° C. and having a low sulphur content. Concentrations of 0·03 per cent. by weight of tri-cresol were found to give an optimum content of inhibitor. The present report deals with a continuation of the investigation on a large scale with prolonged tests in commercial engines. It is shown that benzols stabilised by an inhibitor are little inferior to those purified by acid, and that dilution of unstabilised benzol with petrol retards resinification to a greater extent than would be accounted for by the concentration of unsaturated compounds. The efficiency of various types of oil for absorption purposes has been examined with the conclusion that oils from low temperature and Bergius tars have nothing to recommend them for use in preference to oils from low temperature tar, whilst they have the disadvantage of being more unstable. The variation with temperature of the absorption capacity of creosote and gas oils in use as wash oils is shown graphically on a chart from which the capacity at any desired temperature can be read. Correction tables for application to hydrometer readings and volumes have been prepared so that the correct value at 65° F. can be calculated. Tests have been carried out on the purification of benzols by the Ufer and Instill processes, which aim at

removing those components which cause resinification. Finally, the report contains the annual report of the Engine Tests Sub-Committee for 1928, together with a bibliography of literature relevant to fuels and illustrations by tables, graphs and photographs. W. H. T.

778. MAKING MOTOR FUEL. Standard Oil Co. of California. E.P. 301,450 Sept. 5, 1929. Appl. June 5, 1928. Conv. (U.S.A.) Nov. 30, 1927.

The invention relates to a method for removing sulphur compounds from motor spirits, particularly unsaturated cracked naphthas high in sulphur content, by means of conc. H_2SO_4 at low temperature.

The conventional method of reducing the sulphur content of a cracked naphtha to specification limits has consisted in treating the naphtha with somewhat diluted sulphuric acid at ordinary temperatures, a temperature rise of 100° F. having been frequently experienced. After removal of the acid sludge the naphtha was water-washed, neutralised, and distilled to give end-point spirit. Prior to the distillation the sulphur content was but little altered from the original, showing that non-sulphur-bearing hydrocarbons were removed together with sulphur-compounds in the acid sludge, substantially in the proportions in which these existed in the crude naphtha. Practically the whole desulphurising action was due to polymerisation of sulphur-bearing unsaturated compounds into products boiling above the motor spirit range, and this action was accompanied by corresponding polymerisation and loss of valuable non-sulphur-bearing unsaturated compounds, with detriment to the anti-knock value of the finished spirit. With heavy acid treatment of high sulphur content naphthas this polymerisation loss of valuable unsaturated constituents may exceed 30 per cent.

In the present invention either concentrated or fuming sulphuric acid is employed, the maximum temperature of reaction being limited to a predetermined low value. In this way a selective action is obtained, sulphur-bearing bodies being removed preferentially in the acid sludge, so that major desulphurising is effected prior to distillation, while the polymerisation loss is reduced to about one-quarter of that experienced in the conventional treatment. Moreover, since the *treating* loss, *i.e.*, loss of sulphur-bearing and non-sulphur-bearing constituents to the acid sludge is unaltered by the reduction of temperature, the greater direct removal of sulphur compounds by the acid must be accompanied by a lesser removal of non-sulphur-bearing constituents.

The maximum reaction temperature may be controlled either by precooling the naphtha to a low initial temperature, or by absorbing heat from the mixture during reaction. The latter method is the more economical where the evolution of heat is great. The two methods may be combined, but the initial temperature is unimportant except as a means of controlling the maximum.

The optimum maximum reaction temperature employed for a given reduction in polymerisation loss with a particular naphtha will vary in accordance with the sulphur content of the naphtha and the strength of acid employed. In a particular case for given desulphurising, maximum spirit yield was obtained with a maximum reaction temperature of 15–20° F., while minimum acid was required with 25–30° F. maximum temperature. The true optimum maximum temperature for minimum cost lies between 5° F. and 40° F., and is determined by the balance between the cost of acid, the value of spirit saved, and the cost of refrigeration.

Flow diagrams are given illustrating methods of treatment and cooling for (*a*) batch process; (*b*) continuous con-flow process, with or without multiple stage acid additions; (*c*) continuous counter-current process with heat exchangers. L. H. A.

779. Means for Rendering Combustible Spirit Tanks Proof against Fire and Perforation. Societa Italiana Pirelli. E.P. 301,089. Sept. 26, 1929. Appl. Nov. 23, 1928.

To protect combustible spirit tanks, as used on aeroplanes, against leakage due to perforation, layers of rubber are placed on the inside of the walls of the tanks. One of these layers must be of crude or unvulcanised rubber, easily soluble in spirit. This is protected from the spirit by a covering of metal foil. Further, a layer of fireproof felt is placed inside or outside the walls to minimise the risk of fire after perforation of the tank. The whole of these layers is kept in position by stops of rubber, metal, or metal tubes filled with rubber, which are set between the walls of the tank and the protective sheet of metal foil. J. M. F.

780. Improvements in and Relating to the Refining of Oils. S. Tijnistra. E.P. 318,706. Sept. 12, 1929. Appl. July 12, 1928.

It is the purpose of this invention to control the sulphur content of a finished cracked hydrocarbon oil, in order to avoid an excess of free sulphur, by providing a method of precipitation of the lead compounds produced by the action of the doctor treatment on the oil. The process provides a method which may be continuous and yields a "doctor sweet" final product. The method consists in treating the hydrocarbon oil with doctor solution, separating the treated oil, and then adding to it an aqueous solution of a polysulphide capable of releasing elementary sulphur. The elementary sulphur released is of sufficient amount, such that it only carries out the necessary chemical reaction to precipitate from the oil the lead compounds resulting from the doctor treatment, in the form of lead sulphide. The temperature at which the reaction may be carried out can be as high as 200° F. H. E. T.

Lubricating Oil and Wax.

781. Examination of Transformer Oils. R. Audubert. *Rev. Pétrolifère*, 1929, **340**, 1403-1404.

Cooling oil for transformers must possess, in addition to certain physical properties, a high chemical resistance to oxidation and polymerisation, otherwise the elevated temperature of operation will give rise to deposits of asphaltic bodies, which hinder free circulation and, being acid, are liable to attack the windings.

The quality of a transformer oil is judged according to the following points: Tendency to form deposits; insulating ability; flash pt. and fire pt.; viscosity at various temperatures; loss of weight by evaporation; density; pour point; sulphur content; acid and alkali content; moisture content. It is extremely difficult to obtain oils perfectly free from water, perfectly refined, and of high chemical resistance to oxidation: *i.e.*, minimum tendency to form deposits.

There is at present no rational method of measuring the most important quality of the oil, *i.e.*, its chemical resistance towards oxidation under the actual working conditions. The changes undergone by the oil are a function of both time and temperature. As tests of long duration are inconvenient the temperature must be raised. According to the French specifications the sample is maintained at 150° C. and the amount of deposit soluble in CCl_4 after 5, 50, and 135 hours should not exceed nil, 0·06 per cent. and 0·3 per cent. respectively. It is possible that cracking may commence in the neighbourhood of 150–170° C., but the production of light hydrocarbons

of much less importance than the oxidation of tars to volatile acids which are driven off and not estimated. Such acid products attack the windings and hasten the decomposition of the oil. Since 110–120° C. is recognised as a decomposition point with transformer oils it is preferable to carry out the test of ageing below this point, say 90–100° C., on a sample of several hundred ccs., so that the deposit soluble in CCl_4 will be weighable. When different amounts of sample are used the oil surface exposed to the air must bear a constant ration to the volume if comparable results are to be obtained.

The attempt to approach actual working conditions by the use of copper in the test is open to criticism: the actual ratio of bare copper surface of the windings to the volume of the oil is much smaller in practice than in the test; the ratio varies with the design of the transformer, and cannot by any means be determined. The catalytic action of copper also varies according to the chemical composition and origin of the oil. Furthermore the considerable influence of the electrical field on the oxidation is ignored in all tests. The precise nature of its action is not well known and will vary according to the type and power of the transformer.

The exact conditions of this sludging test vary from one country to another and while the same method will give comparable results on different oils provided all the experimental conditions are rigidly standardised, results obtained by different methods are not comparable, nor are they a true indication of the deposits to be expected in a transformer after a long period at about 90° C. L. H. A.

782. Reclamation of Lubricating Oils (Apparatus for). C. F. Barb. U.S.P. 1,721,328. July 16th, 1929. Appl. Aug. 18th, 1925.

The apparatus is designed especially to reclaim used automobile engine oils. The used oil is charged into a reservoir which is provided with a drain cock for settled matter and an overflow pipe. The overflow from the reservoir discharges through a filter into a settling tank. The oil is then passed to an electrically heated evaporator operated under reduced pressure. When the evaporation of the volatile constituents is completed, the oil is cooled and finally purified by percolation through a filter medium. R. W. L. C.

783. Oil Reclaimer. Charles van Brunt and P. S. Miller. U.S.P. 1,703,648 Feb. 26th, 1929.

Lubricating oil recovered from crankcases of internal combustion engines is constrained to pass down a network of capillary tubes heated at 120–150° C. against an upward current of heated air. It is thus claimed that diluents are removed. A. R. S.

784. Reaction between Lubricating Oils and Phosphorus Pentoxide. C. C. Furnas. *Ind. & Eng. Chem.* (Anal., Ed.) **1**, 185.

In the course of some experimental work, it was found necessary to circulate nitrogen by means of a rotary blower. In order to eliminate oxygen, the gas was passed through a chamber containing yellow phosphorus. When this was installed the blower seized after 15 minutes running. On dismantling it was found that a thick gum similar to shellac had formed on all the friction surfaces. It was apparent that phosphorus pentoxide dust had been carried over into the pump and had reacted with the oil to produce this gum. To verify this qualitative tests were made by shaking up oil with phosphorus pentoxide. Three different standard lubricating oils and kerosine were tried. All gave thick sticky reddish brown gum at temperatures of 30° to 50° C. If water was added to the oil the gum dissolved and would not form again as long as sufficient water was present to form a separate phase. R. W. L. C.

785. Distillation of Petroleum Oils. R. W. Hanna, assr. to Standard Oil Co. of California. U.S.P. 1,729,215. Sept. 24th, 1929. Appl. Oct. 4th, 1923.

The invention relates in particular to the fractional distillation of lubricating oil stocks by means of flashing the oil at a constant temperature, under vacuum, whereby vaporization of the higher boiling constituents is obtained at a reduced temperature, such that cracking is practically eliminated, with consequent improvement in colour of products, raising of the flash point for a given viscosity, and reduction of treating loss. The vapours from the flashing operation are condensed fractionally to produce cuts of the desired characteristics.

The raw stock, preheated to about 400° F. in heat exchangers, is rapidly preheated to 750–800° F. in a heating coil at high velocity and under sufficient pressure to maintain the oil in the liquid phase. This highly preheated oil is released into an evaporating still to which steam may be admitted if desired, and in which any desired vacuum up to 26 ins. mercury may be maintained. This still is heavily lagged, and when unheated a liquid temperature of 630° F. may be maintained. If desired heat may be supplied here, keeping the temperature gradient of heat transfer so small that cracking temperature is not reached in any portion. Vapours and residue are withdrawn continuously, the vapours passing through closed pipes in the lower portion of a fractionating condenser, where the highest boiling fractions are condensed by cooler reflux from above. The condensate is withdrawn and cooled, while uncondensed vapours are passed to a series of fractional condensers maintained at successively lower temperatures, each producing a condensate of desired characteristics. The vapours leaving the last fractional condenser are returned to the middle of the first fractionating condenser, the upper portion of which carries dephlegmating trays and a controllable cooling coil. Condensate is thrown down over the hot tubes conveying the initial vapours, which revaporize the low boiling constituents, forcing them to pass overhead to the final condenser, thereby leaving as residue reflux the lightest lubricating fraction of the operation, freed from flash point lowering constituents.

L. H. A.

786. Refining Oil. T. C. Heisig, assr. to Galena-Signal Oil Co. U.S.P. 1,728,059. Sept. 10, 1929. Appl., May 19, 1926.

The invention provides a continuous method of refining high-viscosity lubricating oils derived from asphaltic or naphthenic base crudes. The stock is diluted with naphtha or other non-viscous hydrocarbon solvent, dried with about 1 per cent. of 93 per cent. sulphuric acid, agitated with about 4 per cent. of 98 per cent. sulphuric acid, and the acid sludge drawn off. The diluted acid stock is mixed with an adsorbent (fine clay, fuller's earth, etc.), to the extent of 1–5 per cent. by weight, and the mixture is pumped to a fire and steam still, where the diluent is distilled off for re-use, live steam being introduced into the oil through sprays up to a temperature of 450–500° F., at which point the fire is drawn and only enough steam maintained to insure agitation. The hot oil is then filtered and is found to be of light colour and great purity.

The invention claims the following advantages as a result of reducing the viscosity by dilution: less acid required to dehydrate the oil; more intimate contact of the main treating acid with the oil; more rapid and complete settling out of the acid sludge after treatment, thereby reducing loss and reducing the amount of clay required to neutralise the acid compounds; and more intimate contact and reaction between the adsorbent and the oil due to the low viscosity and the violent agitation of the distilling process. L. H. A.

787 PROCESS AND APPARATUS FOR MAKING WHITE PETROLATUM. A. Holmes. U.S.P. 1,733,597. Oct. 29, 1929. Appl., Oct. 27, 1925.

The petrolatum-yielding material is treated in the liquid form with fuming sulphuric acid at a high temperature, the residual liquid and carbonaceous bodies are treated with a solvent for the petrolatum, and the purified petrolatum recovered from the solution.

W. S. C.

788. THE VAPOUR TENSIONS OF LUBRICATING OILS IN RELATION TO FLASH POINT AND TO CRACKING. Iselin. *Ann. offices nat. comb. liq.*, 1929, 1, 69.

The invesigation described was carried out in order to determine the relation of vapour tension to flash point and to cracking. For the lighter petroleum fractions, flashing is due to the vaporisation of the more volatile constituents, while in the case of products such as motor and cylinder oils, flashing may be due to vapours formed by incipient cracking of the oil. The study was made with the aid of a cathodic vacuum tube so constructed that the oil contained in it could be heated in an electric furnace and the vapour pressure determined. The gas dissolved in the oil was removed by cautious heating and cooling, and the vapour pressure of the expelled gas determined. This process was repeated until no vapour pressure could be determined. The oil was then heated (2° per min.) and vapour pressures determined until a temperature was reached at which the vapour pressure did not stay constant, when it was assumed that sufficient cracking was taking place for the gases to affect the reading. Corrections were made for the vapour tension of the mercury, the expansion of the mercury column in the furnace, the weight of the column of oil, the capillarity of the oil and the atmospheric pressure. Lubricating oils showed the first decomposition at about 200° C., and the change proceeded rapidly from 260°–300° C. For the spindle oils there was an interval of about 80° C. between the point at which the gas was driven out and the point where decomposition became apparent. This interval diminished with the more viscous oils and did not exist for Pennsylvanian cylinder oil. For oils of flash point (Luchaire) below 200° C. (spindle and machine oils) the vapour tension at the flash point is between 5 and 8 cu. m. Hg. The flash point of motor oils lies in the cracking zone, but in view of their low vapour tension below 250° C., the flash is doubtless due to their own vapour tension. The flash point of cylinder oils (about 300° C.) is undoubtedly affected by cracking, but the flash is mainly the result of normal vapour pressure. By distillation, the flash point could not be increased beyond 370° C., and hence it is concluded that at this point decomposition becomes the controlling factor. It is concluded that the flash is always attributable to vapour tension except with cylinder oils, and here it is a question of how far the two phenomena co-exist. There is thus insufficient ground for changing from flash point to vapour pressure determination; for below 200° C. the vapour pressure is too small to be measured accurately, and when it becomes marked it ceases to be constant.

W. H. T.

789. REFINING OF CRUDE PARAFFIN WAX. J. Y. Johnson. E.P. 320,921. Oct. 21, 1929. Appl., July 19, 1928.

Crude wax is treated in the liquid form with hydrogen at high pressure and temperature. The duration of treatment and the temperature are regulated so that the wax suffers little or no cracking. A catalyst consisting of one or more oxides of the metals of groups 3, 4 or 6 of the periodic system is added and the addition of small quantities of iron, nickel, cobalt or their oxides has an activating effect.

W. L.

790. Cup Grease Manufacturing Methods. H. L. Kauffman. *Refiner*, 1929, 8 (5) 93, 135.

The subject is offered in two parts, and this article deals with formulæ and detailed methods of operation. Cup grease is mainly an emulsion of calcium soap and mineral oil, together with a small percentage of water which aids in preventing the separation of the other two constituents. The soap content varies between 10 and 26 per cent. Much of the success of grease making depends on the kind and quality of the fatty oil used. Horse oil, beef or mutton tallow, lard oil and acidless tallow are the animal oils most frequently used, while cottonseed oil, soya bean oil, corn oil, palm oil, coconut oil and peanut oil are the vegetable oils most generally in use. Mixtures of any or all these can be used with success. Horse oil is probably the fatty oil most frequently used. Stringent specifications for these oils are laid down and typical specifications for lard oil, cottonseed seed oil, horse oil and cake tallow are given.

Various methods are in use for slaking the lime, but in all cases it is very carefully screened after slaking. Too much care cannot be taken in seeing that the lime is well slaked and that it enters the grease kettle in as pure a form as possible. Various methods in use among grease manufacturers are given in detail, together with specifications for the mineral oil used. The fatty oils and the necessary amount of slaked lime are emulsified and heated by steam. When reaction is complete the soap is dried as much as possible by steam heating and agitation. When the soap melts and is smooth throughout, the mineral oil, warmed so that no sudden nor extensive cooling takes place, is added very slowly over a period of several hours. Extreme care is necessary during the addition of the first 20 per cent. of the mineral oil in order to avoid the formation of soap lumps. Oil soluble yellow dye is generally added to improve the colour, and the unpleasant odour is usually masked by the addition of oil of myrbane. Before the completion of the addition of the mineral oil the appearance and consistency of the batch is noted. A rubbery appearance indicates insufficient water, and more must be added; otherwise the grease will separate oil on standing. Additional water must be added slowly and carefully, for the addition of too much water will appear in the finished product as free water.

W. L.

791. Results of Experimental Work in Sweating Slack Wax. H. L. Kauffman. *Refiner*, 1929, 8 (9) 74.

The article commences with a brief resume of the general methods in use for sweating slack wax. Details are given of laboratory experiments in which slack wax received different physical treatment before sweating. The results of these experiments are presented in a series of tables. Slow cooling of the melted wax before sweating is advantageous. The wax yields are consistently higher where the wax was cooled slowly than where the wax was rapidly cooled by ice water.

A higher yield of wax is obtained when the melted wax is slowly cooled till nearly solid, then agitated prior to solidification and before starting sweating, than results by the regular method where the wax is not so agitated before starting the sweating.

W. L.

792. Tests of Lubricating Greases. F. L. Koethan. *Nat. Petr. News*, 21, No. 42, 69 71.

A few rapid observations will often indicate to which class of grease a particular lubricant belongs. Sufficient information may thus be gained to tell whether a grease is suitable for a particular use.

Calcium soap greases have a smooth appearance and lack elasticity They are insoluble in water, melt below the boiling point and some mineral oil usually separates on melting. Under the microscope they are characterised by round globular particle shape. Calcium rosin greases (axle greases) are of similar appearance to calcium fat greases but break down in consistency when stirred or mechanically worked more than the latter. They are insoluble in water and melt above the boiling point of water, but at a lower temperature than the anhydrous soda soap greases.

Anhydrous soda soap greases have an elastic, fibrous nature, the soap being in thread-like strands which may be seen under the microscope if the grease is treated with cold petroleum ether before examination. Sodium soap greases containing water are not often encountered. The water destroys the fibre, giving rounded globules and the mass is a lifeless low-melting paste.

Soda lime greases are mixtures of the two kinds of soap and have a short fibre and high melting point.

Aluminium soap greases are insoluble in water, smooth and often translucent. The oleates are stringy and the stearates elastic. Lead soap greases are smooth, very adhesive and non-elastic. Stear-anilide as a stiffening constituent of grease confers a waxy appearance, a low melting point and much "break." Greases containing it emulsify readily with water. Consistency is probably the most important characteristic of a grease. Tests should be made on the original grease and on the material after it has been worked or broken down by mechanical action. The grease-working device mentioned in A.S.T.M. Specification D127 may be used. The author prefers to use a straight-sided plunger 5 mm. in diameter rather than the tapered cone penetrometer specified by A.S.T.M. The final standard device for the determination of consistence should be of the plastometer type such as that of Bingham. The plastometer gives separate readings for yield values and mobility. Calcium-fat greases have a definite consistency after breaking down which is not reduced by further working. Soda soap greases have very little "break." Calcium-rosin greases have a very great "break" and often re-set on standing for 24 hours.

The A.S.T.M. method (D95-28) is perfectly satisfactory for the determination of water in greases containing ½ per cent. or more of water, but for greases containing only a very small percentage of water, such as a soda soap sponge grease, which should contain less than ½ per cent., the use of 100 c.c. of water-saturated xylol instead of the gasoline gives more accurate results. The xylol is a much better solvent for the soaps and permits all the water to be carried over in a forty-minutes' run, during which time the amount of glycerol carried over is negligible. The addition of fused potassium bisulphate to the mixtures of grease and gasoline in the water determination gives results which agree with the xylol method.

The most satisfactory method of determining the melting point of a grease has been found to be as follows; the grease is pressed into a square loop (side 6·35 mm.) consisting of three turns of 1·59 mm. diam. aluminium wire. The loop is clamped close to the bulb of a thermometer and heated in an air bath. The whole mass drops from the loop within a short temperature range.

The question of the complete analysis of greases is dealt with briefly. For friction tests the machines designed by Herschel and Deeley are recommended.

The resistance of a grease to separation may be determined by applying pressure to a sample contained in a filtering vessel. In such an apparatus a pressure of 5 lbs. per sq. in. caused a separation of 11·6 per cent. of oil from a pressure gun grease in 2½ hours.

Suggestions are also made for the determination of the stickiness or adhering power of greases. A bibliography is appended. R. W. L. C.

793. Grease Making. A. R. Lange. *Nat. Petr. News*, **21**, No. 42, 67-68.

Greases may be classified into the following :—

1. Straight fats or simple blends with mineral oils, pitches, waxes, etc.
2. Greases made by simple neutralisation.
3. Greases made by cold or semi-boil saponification.
4. Greases made by saponification or two-stage reaction.
5. Greases made by substitution of bases.

The only grease of any importance in Class 1 is hot neck grease, which is controlled for melting point by the amount and character of the pitch used.

Class 2 greases comprise those made with rosin or rosin oil and those made with fatty acids. They are prepared by mingling a stream of alkali in solution with a stream of mineral oil containing the rosin or fatty acid. Reaction is very rapid and the greases set very quickly. Axle greases and water pump greases are prepared by this method.

Class 3 includes greases which can be made without employing the customary open kettle boiling necessary for complete or nearly complete saponification. The so-called engine greases and railroad journal compounds may be thus prepared.

Class 4 type of grease includes cup greases, sponge or fibre and brick greases. These greases are made as a rule from fats that are relatively low in free acid content. A neutral soap stock is first prepared by hydrolysing the fat (*a*) in a kettle heated either over an open fire or by a steam jacket, or (*b*) in a closed pressure cooker. The soap is then reduced with the type of mineral oil required in the finished grease. The glycerine produced in the hydrolysis of the fat has a stabilising effect on the grease. Some figures are tabulated showing the time taken to hydrolyse a quantity of fat with different quantities of lime and different amounts of water.

The greases comprising Class 5 are aluminium and lead soap base greases. The aluminium and lead soaps are prepared by interaction of soluble salts of these metals with a soda soap. The resultant aluminium and lead soaps are reduced with the requisite amount of mineral oil to the consistency required. R. W. L. C.

794. Carbon Deposits from Lubricating Oils. C. J. Livingstone and W. A. Gruse. *Ind. and Eng. Chem.* **21**, 904. Oct., 1929.

In three previous communications the present authors have described the results obtained when operating with a single-cylinder engine under laboratory conditions, and have arrived at the following general conclusions : (*a*) That the carbon-depositing tendency of automobile oils can be predicted approximately from the carbon residue test of the oils ; and (*b*) that the carbon residue test is significant probably because it is a rough indication of the volatility of the oils at flame temperatures. In order to confirm these conclusions under the conditions of road operation, observations have been made on a fleet of 18 coaches equipped with sleeve valve engines in actual commercial service. The conditions are such that the engines were running on full throttle most of the time.

The coaches were divided into three groups of six. The engines of one group were lubricated with a paraffin base oil of high carbon residue, well refined and otherwise of high grade. In the second group a paraffin base oil of extremely low carbon residue very drastically refined was used. In the third group a typical naphthene base oil was employed. All three oils were of similar viscosity at 210° F., *viz.* approximately 100 sec. Saybolt.

The following is a summary of the observations: The total distance covered by the six coaches in each group was from 50,000 to 60,000 miles. At each dismantling and inspection, which was carried out at intervals of between 1500 and 3000 miles, a sketch was made of each port showing the area covered by carbon and the shape of the carbon deposit. Each sketch was made on a chart which was a projection of the sleeve port on a horizontal plane. After this the port was cleaned if necessary. When the test had been completed the total port area in square inches closed by carbon for each group of coaches was determined by planimeter measurements from the port diagrams. This figure was converted into a percentage of the total port area per coach covered by carbon during 10,000 miles of operation.

Characteristics of the oils and the results of road tests:—

Oil.	High carbon paraffin oil.	Low carbon paraffin oil.	Naphthenic oil.
Flash, °F.	480	470	500
Fire, °F.	520	535	565
Viscosity at 100° F.	1310	1360	2760
210° F.	105	104	103
Pour, °F.	25	10	20
Colour N.P.A.	4·5	1·5	8
Carbon residue	1·00	0·15	0·70
Total coach miles	51,506	60,540	59,030
Oil consumed (quarts)	2178	2197	2401
Average miles per gallon of oil ..	94.6	110	98·5
Average miles per port cleaned ..	314	401	1556
Area of carbon per coach per 10,000 miles	35·7	29·2	11·8
Per cent. of port area closed with carbon in 10,000 miles	324	266	107

The paraffin base oils produced a carbon deposit which was hard and adhered to the port openings, whereas the naphthene base oil produced a carbon which was soft and fluffy and could be easily removed, and it is considered that the relatively small amount of port closing observed with the naphthenic oil is in part due to the erosion of the deposit by the exhaust gases. The difference in the carbon deposit forming effect of the two paraffin base oils is less than would be expected from the difference in their carbon residue values. Tests with a laboratory poppet-valve engine agreed in general with the results of the road tests. Distillation tests carried out at a pressure of 10 mm. showed that 90 per cent. of the naphthenic base oil was volatile under the conditions of the test at temperatures up to 351° C., whereas only 30 per cent. of the paraffin oils was volatile up to 370° C. when cracking started. In order to further investigate this point the high-carbon and low-carbon paraffin oils were distilled in the same way at 2 mm. pressure. This served to bring out the difference between them. The high-carbon paraffinic oil gave 41 per cent. off between 254° and 373° C. and the low-carbon paraffinic oil gave 49 per cent. off between 271° and 372° C. This difference in volatility is in the same direction as the difference in carbon formation in the coach engines.

In a general summary of the work of the authors on this question it is concluded the Conradson carbon residue test is a fairly reliable index of the carbon-forming tendency of the oils in a poppet-valve engine. Of seventeen oils studied, fifteen gave fair agreement between carbon formed and carbon residue test in a straight line relationship. The agreement is more conspicuous with paraffin base oils. For sleeve valve engines a paraffinic base oil of very low carbon residue was only slightly better than one of normal carbon residue value, and both were strikingly inferior to the naphthenic oil. R. W. L. C.

795. Iodine Values of Mineral Oil Products. I. B. M. Margosches, B. Krakonetz, and F. Schnabel. *Petr. Zeit.*, 1929, 25 (1179-1186).

Previous work on iodine values of mineral oils is described with special reference to the methods of Hübl, Wijs, Hanŭs, and Winkler. The conclusions derived from experimental work concerning these methods are given in the following:

I. The rapid method for iodine values can be applied successfully in the following modified manner to all those mineral-oil products which dissolve and emulsify properly, especially cracked benzines and higher-boiling asphalt-free mineral-oil products.

1. Acetone, ether, ethyl acetate, mixtures of acetone and ether (1/2 or 5) or of ether and alcohol (1/2 or 5) are applied as solvent for the experimental substance.

2. To obtain the requisite iodine excess of 93–95 per cent. one adds to cracked benzines (0.1 gms.) 25 or 50 c.cm. of $\frac{N}{2}$ or $\frac{N}{4}$ alcoholic iodine solution and 25 c.cm. of $\frac{N}{5}$ alcoholic iodine solution to a modified quantity of high-boiling products.

3. After adding about 200 c.cm. of water and waiting five minutes (blank experiment carried out under similar conditions), the values obtained by the Hübl method were very concordant.

II. It is impossible so far to obtain iodine values by the rapid method for heavy and asphaltic mineral oil products.

III. 1. Hübl's method for cracked benzines yields, after increasing the time from 24–72 hours, somewhat constant saturation values, but increasing saturation values are obtained with high-boiling mineral oil products.

2. The iodine values, according to Hanŭs' and Wijs's methods for cracked benzines, are much greater than Hübl's values, and this is attributed to increased substitution of halogens, especially as the latter are never regarded as absolutely free from substitution.

3. Winkler's method produces up to the present rather abnormal values which still need confirming. W. S. C.

796. Process for Dewaxing Oils. L. W. Naylor. U.S.P. 1,722,307. July 30, 1929. Appl., June 7, 1927.

This invention relates particularly to the separation of mineral wax, such as paraffin from a mixture of such wax and petroleum oils. The method of dewaxing consists in shock chilling solutions of long residuum or amorphous wax petroleum oil cuts in a suitable solvent (*e.g.*, naphtha) in presence of finely divided litharge. The oil-wax naphtha solution acts like a colloidal solution at temperatures near the coagulating point of the wax. The finely divided wax, which separates on chilling, coagulates on nuclei consisting of particles of litharge. The heavy composite wax-litharge particles separate readily and completely from the oil-naphtha solution. The latter is drawn off and the oil recovered by topping. The litharge is recovered and used again. Various other materials have been tried in place of litharge, but with less success. Five per cent. by volume of litharge has been found to be a sufficient quantity. The physical state of the material has some influence on the success of the process. R. W. L. C.

797. Method of Stabilising Clay Treated Oils. G. F. Olsen. U.S.P. 1,724,510. Aug. 13, 1929. Appl., Aug. 31, 1928.

This invention relates to a process for stabilising mineral oils which have been decolorised and neutralised by treatment with absorbent clays. Deleterious odorous volatile substances and other volatile substances lacking in colour stability are removed by atomising the hot filtered oil from the clay treatment process with steam into a non-oxidising atmosphere. The objectionable volatile bodies are completely removed and a stable oil remains. R. W. L. C.

798. Recovery of Entrained Oils from Clay. G. F. Olsen. U.S.P 1,720,144. July 9, 1929. Appl., Jan. 22, 1927.

The invention relates to the recovery of a valuable quantity of lubricating oil which remains entrained in the cakes of clay obtained during the contact filtration in filter presses of mixtures of petroleum oils and pulverised clays or other absorbent bodies. The method of recovering the lubricating oil from such filter cakes consists in forcing through the cakes, in place in the press in which they were originally formed, a less viscous oil in sufficient quantity to displace the entrained oil from the cakes. The less viscous oil can be used repeatedly, and by mingling the effluent oils a lubricating oil of intermediate viscosity can be produced. R. W. L. C.

799. Solubilities of Oils and Waxes in Organic Solvents. J. W. Poole. *Ind. and Eng. Chem.*, 1929, 21, 1098.

Data concerning the relative solubilities of wax and oil in several organic solvents have been secured for the purpose of encouraging the commercial use of the extraction process for the separation of petroleum oils and waxes. A large variety of solvents was chosen with the view of covering fairly well the different types that are commercially available, and from the preliminary results the seven solvents that were chosen for further work were those that seemed most likely to give the maximum information.

Acetone: Although showing a much higher solubility for oil than wax, it is so poor a solvent for either that its use for extracting oil from oil-wax mixtures will probably be limited to laboratory work.

Butanol: This has exceptionally good selective powers. Even as low as 0°C. it dissolves practically one-third its weight of oil, while up to 25°C. the solubility of wax is below 2 per cent. About 35°C. the solubility of wax increases very fast, and at 50°C. has reached the point of complete miscibility.

Butanol-Acetone (50 : 50) Mixture: The results with this mixture indicate no advantage over butanol.

Ethyl Acetate: This shows a very good selective action. Except for the fact that up to 25 to 30°C. oil is considerably less soluble than in butanol, and that the decided increase in the solubility of the waxes comes at a lower temperature than for butanol, the characteristics of these two solvents are quite similar.

Butyl Acetate, Chlorobenzene, Toluene: Except for butyl acetate there appears little possibility of these solvents being of any use. Butanol offers the best possibilities for solvent extraction, when one considers selective solvent power, temperature of use, volatility and initial cost. W. L.

800. Solvent Extraction of Paraffin Wax. J. W. Poole. *Oil and Gas J.*, 1929. 28, (19) 99, 185.

The article commences with a brief survey of the production and treatment of "paraffin-distillate." The disadvantages of wax separation as at present practised are pointed out.

It is said of lubricating oil manufacture that, due to abundance of raw material and in spite of wasteful methods, a class of lubricants 75 per cent. satisfactory have been produced, but there is a need of better lubricants, and any process that will produce equally good lubricants cheaper, or better lubricants at a cost commensurate with their value, has a most attractive future.

The author considers it probable that some selective solvent may be found and lists some of the advantages that would accrue from a selective solvent extraction process. Certain requirements that such a solvent must meet in order to be of use are:

1. It must selectively dissolve, without permanent combination, one or other of the constituents of paraffin distillate and must have a sufficiently high solvent power for this constituent to avoid the use of excessive volume of solvent.

2. At temperatures not excessively elevated above those of separation the solvent must have sufficiently high solvent capacity for both wax and oil to dissolve completely such mixtures as may be encountered in practice.

3. The solvent must be of such a nature that it can be removed from both components of the oil-wax mixture, and the removal must consist not only of a purification of the product, but of a recovery of the solvent.

4. The solvent must establish such physical differences between the two portions of the mixture that the two may be easily separated.

5. The solvent must effect no adverse change of condition of the oil or wax.

6. The solvent should be easily separated from water, or such moisture content as cannot readily be removed should have little effect on its solvent properties. W. L.

801. Relations between Physical Characteristics and Lubricating Values of Petroleum Oils. E. D. Ries. *Ind. and Eng. Chem.*, Anal. Ed., **1**, 187.

The author discusses the significance of the routine tests usually carried out on lubricating oils in relation to lubricating value. The following observations are made:

Gravity and colour are unimportant.

The numerical values of the A.S.T.M. cold test do not indicate the temperature at which an oil will cease to flow under condition of pump lubrication. For example, by applying suction to a ¼-in. diameter glass tube immersed 2 in. beneath the surface of oils, it was found that oil could be sucked up even when the temperature was reduced 10 to 16°F. below the A.S.T.M. cold-test temperature of the particular oils (see also Wilson, Oak and Barnard, *S.A.E. Jour.*, **22**, 213).

The carbon forming tendency of lubricating oils as determined by the Conradson test is considered to have a real significance. Pennsylvanian oils are often stated to give more carbon than others of the same viscosity owing to their wax content. In order to determine this point, the carbon residue values of some Pennsylvanian distillate fractions have been determined both before and after dewaxing. The values for the dewaxed oils are, in general, slightly higher than those of the waxy distillates.

Flash-point and fire tests give some indication of the volatility of an oil, but are not a true measure of the average volatility. A much more suitable test would be a vacuum distillation assay, such as that of Peterkin and Ferris (*Ind. and Chem. Eng.*, **17**, 1248). The temperature-viscosity coefficient is probably of equal importance to actual viscosity. It is pointed

out that slope of the log. temperature *versus* log. viscosity curve can be conveniently expressed as the ratio of the viscosity at 100°F. to that of 210°F.

The temperature-viscosity coefficient of lubricating oil is not necessarily dependent on the crude oil source as this coefficient can be changed by altering refining methods. High vacuum distillation methods tend to produce oils with flatter viscosity curves.

The question of "oiliness" and resistance to oxidation are also discussed.

R. W. L. C.

802. Distillation Process. J. C. Ryder, Assr. to Petroleum Derivatives. U.S.P. 1,727,380, Sept. 10, 1929. Appl., Jan. 5, 1924.

The invention provides a process for the continuous distillation of mineral oils under vacuum without substantial decomposition. A small stream of oil flowing in a large inclined passage is subjected to a progressively increasing degree of heat, and the resulting vapours are allowed to escape from the free surface continuously as formed. The vapours are free to flow both in the direction of and counter to the direction of oil flow, and are drawn off at intervals along the course of the stream. The vapours from each interval are separated from liquid at reduced velocity in towers without material refluxing, and are then fractionally condensed. Receiving tanks for distillates and residuum are provided in duplicate with the usual connections and valves for oil, vacuum, and air.

It is claimed that the process may be made to yield high viscosity lubricants with a minimum of objectionable cracked constituents by direct overhead distillation.

L. H. A.

803. Iodine Numbers of Lubricating Oils. W. F. Seyer and J. S. Allen. *Oil and Gas J.*, 1929, **28** (14), 48, 141.

The determination of the iodine numbers of a large number of lubricating oils before and after use in automobile engines was undertaken with the object of determining what structural decomposition, if any, the oil undergoes. The fact that a change in the oil as a whole might be brought about by the absorption of unsaturated compounds out of the gasoline and by dilution with heavy-ends was not overlooked. A modified Hübl method was used in which 0·35 gr. of dried oil was placed in a flask, dissolved in 10 cc. carbon tetrachloride and 25 cc. iodine monochloride solution added. The flask was surrounded by melting ice and placed in the dark for 2 hrs., after which 20 cc. potassium iodide solution (10 per cent.) and 100 cc. water were added. The excess iodine solution was titrated with standardised thiosulphate solution using starch indicator at the end. Samples of oil were drawn after definite milages from a large number of cars. The diluent was not removed, this having the effect of raising the iodine number of the oil owing to the large amount of unsaturateds in the gasoline. All samples of oil were dried over calcium chloride for one week and each sample centrifuged before use. The results show, with very few exceptions, that the iodine number decreases in spite of dilution, the decrease being greatest during the first 500 miles. The oil from cars using oil filters showed similar results to the others. The general conclusion is that the unsaturated hydrocarbons being more easily oxidised than the saturated are removed during combustion by conversion into gaseous products, or they may be only partially oxidised to form the insoluble sludge and removed by precipitation.

H. S. G.

804. Sodium Peroxide for the Determination of the Condition of used Insulating Oils. G. Schwittay, *Petr. Zeit.*, 1929, **25** (6–8).

It is essential to ascertain quickly whether a used insulating oil is strongly acid or tarry, and still usable or unusable. The use of sodium peroxide

as a means of determining these criteria is recommended. It must be prepared in a special way, either Solution 1: Equal parts of N/10 sodium carbonate and N/10 caustic soda solutions are mixed, 100 c.cm. of the solution taken, 2 gr. of sodium peroxide added slowly, and allowed to dissolve, or :—

Solution 2. 4 gr. of sodium peroxide are added slowly to 100 c.cm. of distilled water, and allowed to dissolve. The solutions are now heated in a drying oven to 80° C. for ½ hr., and are ready for use after cooling. Experiments were carried out on five different oils from used transformers, *e.g.*

	a	*b*	*b*¹	*c*	*d*	*e*	*f*	
Acid no. ..	0·336	0·78	0·78	2·91	4·03	0·78	0·22	mg.
Saponification no. ..	—	—	—	5·88	7·84	—	—	KOH
Colour of oil ..	reddish-brown			yellow		reddish brown	yellowish brown	

The acid number and colour in transmitted light were established, and saponification numbers for *c* and *d* determined. Results show that the sodium peroxide causes the tar and naphthenic constituents to separate. Solutions 1 and 2 were separately added to a dark red oil. The oils turned greyish-brown and there was no separation after 12 hrs., but when both samples were placed in a water bath for 45 mins. the greyish-brown colour vanished and the oils began to clarify. Solution 1 became bright yellow and the separation oil/solution was quite significant, and it became turbid at room temperature. Solution 2 produced a yellow coloration, but not so deep as Solution 1. On heating a black substance separated which turned bown suddenly when cold, and whitish grey needles were precipitated. The separation amounted to 4·6 per cent. by volume, and remained very distinct. Thus the separation of the oil with oxidation products appears to be almost proportional to the saponification number and not proportional to the mechanical impurities. Separation of oil and solution by diluting with benzene in the cold and placing the sample in hot water, is greatly accelerated. It was found that the worse the oil the quicker the separation, and the better the oil the slower the separation. W. S. C.

805. Lubricating Oils. H. M. Wells and J. E. Southcombe. E.P. 320,899, Oct. 21, 1929. Appl., July 17, 1928.

Synthetic hydrocarbons, manufactured by the hydrogenation of carbonaceous materials, are treated by the addition of small quantities (1 per cent. or less) of organic acids containing more than five carbon atoms.

W. L.

806. Revivifying Absorbent Materials. W. M. Stratford, Assr. to The Texas Co. U.S.P. 1,724,531, Aug. 13, 1929. Appl., Feb. 11, 1925.

Clays and absorptive materials may be regenerated by extraction with suitable solvents. Spent Fuller's earth may be regenerated to nearly its original efficiency. Cold light petroleum extracts only a little asphaltic matter and does not revivify the earth although the material extracted by acetone, which does revivify the earth, is mainly soluble in naphtha. If the used clay is extracted with naphtha at 600° F. and 150—500 lb. pressure it is revified to almost its original activity. The necessary temperature and pressure at which to use hydrocarbon oils for extraction purposes has to be determined by experiment. T. A. S.

807. Contribution to the Knowledge of Mineral Oils. J. Tausz and M. Dreifuss. *Petroleum*, **24**, 1383–1412.

The authors discuss the work of Dallwitz-Wegener and others on the contact angle of lubricating oils and metallic surfaces. The results of determinations made with the Dallwitz-Wegener universal oil tester (*Petroleum*, **16** (1920), 249) on transformer, gas, train, castor, turbine and cod oils are given. The method of using the apparatus consists in the determination of the height to which the oil will rise in a capillary tube, (*a*) from a small metal capsule and (*b*) when the oil is placed between two horizontal metal discs, the upper disc being just pierced by the capillary tube. The latter condition gives the lower capillary rise. The difference between the two capillary rises enables the wetting tension of the oil for the metal surfaces to be calculated. The use of discs of different and dissimilar metals showed that the nature of the metal did not affect the result. The authors consider that the contact angle between the oil and the metal surfaces cannot be determined by such means.

The work published on the surface tension of lubricating oils is reviewed. The methods for determining this characteristic based on the weight of a drop are unreliable as the results are influenced by the viscosity of the oils. The surface tensions of oils as measured by the capillary rise method between 20° C. and 100° C. bear in general a nearly linear relationship to the temperature.

It is further shown that the calculation of the molecular weight from the surface tension measurements according to the equation of Eötvös, Ramsay and Shields is not possible for the viscous mineral oils as the values of Ke in this equation are not constant for such substances.

If the molecular weight is known, an approximate value for the critical temperature can be calculated by means of the above value Ke. It is possible to calculate the boiling points of oils which cannot be determined directly from the critical temperature.

The ration Y20/M for different oils has been calculated and compared with the dielectric constant. In general, this ratio decreased with increasing dielectric constant.

In viscosity estimations in the smallest possible capillaries the Poiseuille equation loses its validity, but the equation is still valid for the oils investigated even when the thickness of the oil film was only 0·003 mm. The viscosity estimation was carried out for very small amounts of oil (a few cubic millimetres are sufficient) by determining the rate of flow between highly polished optically flat metal surfaces. The system metal-oil-metal constitutes an electric condenser and the time of outflow of the oil is estimated by means of electrical measurements depending on the alteration in the capacity of the dielectric.

A series of experiments were carried out with a bronze slider on a steel plate using both mineral oils and castor oil and employing electrical measurement of the thickness of the oil film. The results showed that with oil films of the same thickness doubling the tractive force more than doubled the sliding velocity.

A comparison of the values for the dielectric constant K and the refractive index n_d for a number of mineral oils has been made and it is suggested that a mineral oil is more valuable when it possesses a small numerical value for $\sqrt{K} - n_d$ and a high viscosity. R. W. L. C.

808. The Set Point of Some Mineral Oil Mixtures. J. Terpugoff. *Petr. Zeit.*, **1929**, **25**, 1213–1214.

Deviations between the observed and calculated set points of mineral oil mixtures may be positive or negative according to the nature of the original

oil. Positive deviations occur with certain blends of Russian, Texas and Pennsylvanian oils, and negative deviations are experienced with certain blends of Russian, Tex and Mid-continent oils. In some cases the set point of the mixture is lower than those of the initial oils. Since mixtures of oils of the same origin give differences between observed and calculated set points which lie within the limits of experimental error, it follows that mixtures of oils of various origins undergo a change in their properties. With positive deviations some oils are rendered more viscous although there is no change in viscosity at 20°, 50° and 100° C.; with negative deviations some oils are rendered more liquid. The greatest deviations occur with Texas oils which become more liquid with increase of temperature. Thus those oils whose viscosities and set points are decreased in mixtures, change their liquid state very quickly with increase of temperature. It is assumed that, say, at 20° C. some hydrocarbons of those oils are only present in the colloidal state and with increase of temperature, or on mixing with oils of other origins, these hydrocarbons pass into ordinary solutions.

W. S. C.

809. Process for Producing Lubricating Oil from Petroleum Oil. J. B. Terry and R. A. Halloran. U.S.P. 1,721,719. July 23, 1929. Appl. Feb. 14, 1923.

This invention relates to a process for the production of libricating-oils and other products from the asphaltic residue obtained by the distillation of crude petroleum.

The method consists in destructively distilling the asphalt to form a substantially asphalt free residue having a melting point of higher than 200° F. and a solubility of not more than 65 per cent. in 86° Baumé gravity gasoline. The distillation may also be carried out to produce a brittle non-sticky pitch or a mixture of intumescent material and viscous oil. These residues are then extracted with a solvent (petroleum naphtha) which is capable of dissolving the viscous oil, but which is not a solvent for the remaining constituents of the residue. The viscous oil is obtained by evaporating the extract.

R. W. L. C.

810. The Production of Transformer and Switch Oils. Dr. Typke. *Petr. Zeit.*, 1929, 25 (819–824).

After removing the paraffins from crude petroleum, *viz.* Russian oils, Venezuelan and Pennsylvanian oils, the distillate whose viscosity lies between 3° and 15° E, and whose flash point lies between 120° and 200° C. is utilised to produce transformer and switch oils. Sulphuric acid, lyes and bleachers are applied in the refining. 93–94% H_2SO_4 is used which removes unsaturated and asphaltic substances, and many oils of an acid nature; bleachers adsorb acid and asphaltic compounds. The Edeleanu refining process is used to a great extent in Germany. This makes use of the action of sulphurous acid which produces sparingly soluble paraffin and naphthenic hydrocarbons, and a very soluble extract of unsaturated, oxygen-containing and sulphur compounds. Those oils which are subjected to a more intensive H_2SO_4 refining contain certain protective substances which are extracted and hinder the oxidation of the oil. A less intensive refining with acid is applied in most countries.

W. S. C.

811. Removing Amorphous Wax from Petroleum Oils. P. M. Travis, assig. to Travis Process Corp. U.S.P. 1,732,143, Oct. 15, 1929. Appl. July 16, 1927, and U.S.P. 1,732,144, Oct. 15, 1929. Appl. July 16, 1927.

Under the first patent the oil is blended with a diluent, heated to approximately 100° F. and agitated to ensure complete solution of the wax, and then

gradually cooled to about -10° F. in the presence of a finely divided solid. The chilled oil is then subjected to centrifugal pressure to effect the separation of the wax and solid from the chilled oil. The mixture of wax and solid is continuously discharged from the zone of centrifugal action at one point with movement toward the axis of rotation while the wax free oil and naphtha is discharged continuously in a similar manner at another point.

In the second patent the oil is given a preliminary treatment to remove asphaltic constituents and other impurities which act as protective colloids and is then blended with a diluent and treated as in the previous patent with the exception that no solid material is added.

W. L.

812. Obtaining Lubricating Oils and Phenols from Coal Tar. A. Weindel. U.S.P. 1,726,638, Sept. 3, 1929. Appl. Dec. 12, 1924.

The viscous oils present in low-temperature tar tend to become decomposed and to lose their viscosity in consequence of the high temperatures of distillation. The patent describes a process which is designed to overcome this difficulty in the following manner. The tar is distilled up to about 240° C, and a quantity of benzene is added to the residue in order to separate insoluble asphaltic materials from the residue. The benzene is removed by distillation from the resultant solution, and a water-miscible solvent is added to the lubricating oil material. Water is then mixed with the solution whereby the oil is caused to separate out and the phenols to remain in solution in the aqueous layer. Acetone is quoted as being a suitable water-miscible solvent.

W. H. T.

813. The Determination of Oil in Paraffin Wax. D. P. Weld. *Oil and Gas J.*, 1929, 28, **19**, 44.

Methods used for the determination of oil in paraffin wax all suffer from the lack of distinction between the properties of oil and wax, as well as from the variations of the properties of oil and wax derived from different sources. The press method for the estimation of oil and water, although the oldest in use, is unsuitable because of the impossibility of obtaining concordant results and the lack of direct relationship to the actual oil content of the wax. Several methods have been devised in which a more or less sharp separation of oil and wax is obtained by the use of solvents such as alcohol-ether mixture, ethyl acetate, benzene, light gasoline and nitrobenzene. They are, however, all open to the objection that although all the oil can be extracted from the wax, a portion of the latter is invariably carried into the oil extract. The well-known Holde method is tedious and inconvenient because of the necessity of working at low temperatures. The method of Wyant and Marsh consists of the precipitation of wax from an acetone solution at 60° F. If the melting point of the precipitated wax is too high, it is an indication that the extraction has removed too much wax along with the oil. On the other hand, a low melting point indicates incomplete extraction of the oil. Like Holde's method, that of Wyant and Marsh is not suitable for waxes containing small percentages of oil. Henderson and Ferris, using a special type of apparatus, effect the extraction of oil from wax with nitrobenzene. The solvent and sample are mixed at 70° C, and the mixture cooled to 32° C. The nitrobenzene-oil solution is removed and the process repeated. The purified wax is finally weighed and its melting point determined. This method is satisfactory when applied to slack waxes containing upwards of 35 per cent. of oil, but the authors do not recommend it for waxes containing small amounts of oil.

Wilson and Wilkins' method depends on the determination of refractive index of the sample and the reference of the result to previously constructed refractive index—wax and oil content curves. The weakness of the method lies mainly in the fact that it is necessary to know the refractive index of the oil in the sample and of the wax extracted with the oil. The universal application of this test would require the compilation of refractive index data for all crudes. In Diggs and Buckler's refractometric method the composition of oil wax mixtures is determined by referring the refractive index of the mixture to a curve showing the relationship between refractive index and composition. The method is similar to that of Wilson and Wilkins, but the principal feature is the way in which the refractive indices of the oil-free wax and wax-free oil components are determined. The Solidification Point Method makes use of an oil-wax residue which is obtained from the sample by extraction with ethylene dichloride. The oil-wax residue is dissolved in a weighed amount of a reagent oil of known solidification point, and the solidification point of the mixture determined. The percentage of wax in the mixture of reagent oil and oil wax residue is obtained by referring the solidification point of the mixture to a curve and calculating the oil content from the reading of the curve. The A. S. T. M. has recently adopted as a tentative method a modified form of the old press method, which, although not giving true evaluations of oil content, indicates definitely the degree of refinement of the wax. At present there is no method whereby the actual oil content of paraffin wax can be determined with ease and accuracy. W. H. T.

814. Consistency of Soap Thickened Oils (Measuring Methods). D. P. Weld and M. F. Pratt. *Nat. Pet. News*, **21**, 42, 73–76.

The construction of a modified form of the Herschel and Bulkley burette consistometer (*Ind. Eng. Chem.*, **19**, 134 (1927)) is described. The apparatus consists of a burette, surrounded by a water jacket, and fitted with screwed connections so that capillary tubes of different internal diameters can be attached.

The distance from the bottom of the burette to the zero mark and to each graduation corresponding to each multiple of 5 c.c. is accurately measured before assembling the apparatus. These measurements, together with the lengths of the capillaries, are necessary for calculating the mean hydrostatic head. The apparatus was operated at 75° F.

The capillary tubes were calibrated by making runs with four oils of known viscosity. The mean hydrostatic head was calculated by means of Meissner's approximation formula. The general procedure used in making a run was exactly as described by Herschel and Bulkley (*loc. cit.*).

The time of outflow of each 5 c.c. portion of liquid was measured by the alternate use of two stop watches. The calibration data obtained were plotted. The curves drawn through the groups of points for the four oils used for the calibration are straight lines passing through the origin.

The diameter of the capillaries used was calculated from the well-known equation for the stream line flow of a viscous liquid neglecting the term involving the correction for kinetic energy. Reference should be made to the original for mathematical formulæ and method of calculation, and for graphs showing (*a*) the pressure-rate of flow curves of soap thickened oils made from white rose oil and ½ to 4 per cent. of saponified fat, (*b*) relation of stiffness of soap thickened oils to viscosity of oil base, (*c*) the stiffness in dyne-seconds/cm² of soap-thickened oil versus saponified fat content, (*d*) relation of yield shear value in dynes per sq. cm. of thickened oils to saponified fat content.

The consistometer has been used with good results for control of manufacturing operations. A batch containing too little oil is made up as a rough

match for the desired product. A sample of this batch is tested and the estimated amount of oil added and the sample retested. Whence an accurate estimate of the amount to be added to the whole batch can be made.

R. W. L. C.

Crude Oil.

815. Determination of the Presence or Quantity of Water in Oil Tanks and the like. A. B. Abbott and R. Abbott. E.P. 320,581. Oct. 17th, 1929. Appl. Jan. 19th, 1929.

A testing mixture is used comprised of:

		pts.
Oil resisting water soluble ingredient ..	Treacle	32
Water absorbent ingredient	Chalk (in granular form) ..	32
Binding ingredient	Jeweller's rouge	2
Preserving ingredient	Turpentine	0·166
Colouring ingredients	Sulphurated Potash Solution (10% in water)	0·166
	Tincture of Ferric Chloride ..	0·083

A thin even coating of this mixture is applied to a dip rod which is lowered vertically into the tank until its lower end rests on the bottom. It is kept in this position for from five seconds to five minutes depending on the nature, *i.e.*, viscosity, or density, of the oil. When removed the presence and depth of water in the tank will be indicated by a lightening of the colour of that part of the composition that has been immersed in water. In the case of heavy or dark coloured oil the oil adhering to the rod is removed by a rubber scraper and at that part only of the rod which was immersed in water the composition will be removed also.

W. L.

816. Breaking of Emulsions of Hydrocarbon Oils and Water. C. Arnold. B.P. 319,623. Appl. Mar. 24th, 1928.

The emulsions concerned are stable suspensions of water in crude oil, *i.e.*, "cut oil," bottom settlings, pond slops and water-gas tar emulsions from gas-works. Breaking of the emulsions is effected by adding a water-soluble sulphonic acid formed by the action of concentrated sulphonic acid on petroleum lubricating oil, or a material containing such sulphuric acid, and keeping the emulsion at an elevated temperature. Water-soluble sulphonic acids are the principal organic constituents of sludges formed by the treatment of petroleum lubricating oils with fuming sulphuric acid or sulphuric anhydride; they are also found in admixture with other organic products in the sludges obtained by the treatment of such oils with conc. H_2SO_4, not fuming. The H_2SO_4 is suitably removed. The author claims that it is best to add 5 per cent. by weight of the actual sulphonic acid, or a small fraction of 1 per cent. by weight for usual crude oil emulsions. The proportion to be added depends upon the stability of the emulsion and the minimum required can be determined by experiment. The emulsion is heated either before or after addition of acid above 150° F. A method is also recommended in which 5 per cent. by weight of an unneutralised sludge containing water-soluble sulphonic acid is added to the emulsion and heated above 170° F.

W. S. C.

817. Treatment of Crude Oils, Tars, Bituminous Residues and the Like. L. Bolgar. E.P. 319,718. Sept. 23, 1929. Appl. Mar. 23, 1928.

A process is described for the production of a composition of matter, *e.g.*, ebonite-like substances. Sulphuric acid, waste acid, or acid sludge (25 per cent. by volume, calculated for concentrated acid) is mixed at equal temperatures between 180° C. and 300° C. with crude oils, tars, or bituminous residues. Stearin pitch is added before or during the acid treatment, further heating is carried out, and the resulting mixture is mixed with fillers such as sand, stone meal, steatite meal, asbestos, fibrous substances, etc. The final homogeneous mass is pressed into moulds to form such objects as electric insulators, pipes, cable shafts, roof coverings, etc. The acid-treated raw materials are treated with dilute solutions of bases, and fluxing agents may be employed. W. H. T.

818. Method of Breaking Down Crude Oil Emulsions. W. Geritz. *Pet. Times*, 1929, 22 (559), 615.

In the method described, use is made of the crude naphthenic acids obtained by the distillation of Boryslaw petroleum. The reagent in dilute form is introduced into the crude oil pipe line close to the well head and before the crude enters the gas separator. The oil and reagent are mixed on falling into the gas separator and pass through a pipe to the bottom of a production tank. A T-piece outlet is attached to the end of the pipe in order to obviate the shock produced by the liquid dropping vertically. The liquid enters the tank, where it is maintained at 28–32° C. in order to ensure fluidity of the oil and to allow of separation of water particles. The formation of vapours capable of causing distillation of the lighter portions of the crude is avoided by placing the steam pipe at the bottom of the tank. The emulsion entering the bottom of the tank encounters separated water, and, on mixing with it, contributes by coagulation to the separation of water particles. Entering the emulsion at the bottom obviates the necessity for agitating the upper layers of oil and reduces the distance which a bubble of oil has to cover in comparison with its entry from the top of the tank. W. H. T.

Fuel Oil.

819. Relationship between Calorific Value and Other Characteristics of Residual Fuel Oils and Cracked Residuums. W. F. Faragher, J. C. Morrell and J. L. Essex. *Ind. and Eng. Chem.*, 1929, 21, 933.

The authors find that the relationship between calorific value in B.T.U. per lb. and the A.P.I. gravity of cracked residuums is linear. They confirm also the linear relationship for uncracked fuel oils that have been reported by other investigators. Formulæ for calculating the calorific value per lb. of dry oil are given as 17,010 + (90 × A.P.I. gravity) for straight run fuel oils and 17,645 + (54 × A.P.I. gravity) for cracked residuums, The calorific value per gallon may be obtained by multiplying each equation by the weight of a gallon of the oil. The formulæ are based upon bomb determinations of the calorific value of a large variety of fuel oils and cracked residuums.

The source of the oil, the B.S. material, the volatility, etc., do not appear to affect the calorific value so long as the A.P.I. gravity is not altered.

The average carbon to hydrogen ratio for straight run fuels is 7.08, for normal residuum 8.66 and for flashed residuum 9.22. B.S. material separated from a flashed or normal residuum contains organic matter, soluble in organic

solvents such as chloroform, carbon tetrachloride, carbon disulphide, aniline, etc., to the extent of 40–45 per cent. The dissolved matter has the same carbon-hydrogen ratio as the residuum itself. There is present, however, about 55 per cent. of insoluble material containing about eighteen times as much carbon as hydrogen. The ash content is high and the sulphur content fairly high. W. L.

820. Dehydration of Moist Fuels and Liquid Heavy Hydrocarbon Oils. I. G. Farben. Aktien. E.P. 317,771. Aug. 16, 1929. Appl., Apr. 16, 1929.

The patent describes an improvement of the invention claimed in E.P. 274,858 of July 21, 1926. Heavy fuels or oils or bituminous solid fuels are heated under pressure in order to cause their contained water to separate out as a lower layer. The treatment is preferably carried out in an autoclave at temperatures between 100° and 200° C. after the addition of salts and other substances such as aniline, brown-coal-tar oils, etc. By means of these additions, the separation of the water is accelerated. W. H. T.

821. The Heat Capacity of Oil Products at High Temperature. Tichomirov and Juze. Neft. Khoz, 1929, 1, 74.

In designing distillation or cracking plants, a knowledge of the heat capacity of oils at high temperatures is essential. The existing standard method of calorimetric determination does not give direct values of heat capacities at high temperatures, the main difficulty being the radiation loss. After reviewing other workers' methods of determination, the authors describe their procedure in detail with the aid of sketches and tables. The outer surface of the calorimeter is heated by vapours of acetone (b.p. 57° C.), alcohol (b.p. 78° C.), water, xylol (b.p. 139° C.) or naphtalene (b.p. 218° C.), with the advantage that the surface of the calorimeter can be easily maintained at a particular temperature. The measurement of temperature is by a Wheatstone bridge method and the formulæ for correction are given. The results obtained by this direct method check fairly well with those given by the empirical formula of Fortesch and Whitman and by the method of Karavaev, the comparison being shown by means of curves. W. H. T.

Special Products.

822. Recovering Litharge from Sludge. *Oil and Gas J.*, Oct. 24, 1929, **46**.

It is well known that sodium plumbite ("doctor") has been used for many years in the treatment of sour or sulphur-bearing oils. This treatment has proved to be one of the most efficient and simplest processes for sweetening gasoline. The simplicity in handling the reagent and the fact that it does not require skilled technical supervision is much in its favour.

Doctor solution as it is usually prepared consists of two parts of caustic soda, 16 parts of water, and one part of litharge. The chemicals are placed in a suitable mixing tank where they are agitated with air for a few minutes, then allowed to settle. After the excess litharge has settled out the clear liquid is drawn off and is ready for use. Usually a small quantity of sulphur is added to complete the sweetening process. Frequently the colour of the gasoline is improved in the process, especially in the treatment of gasoline from cracking plants. Formerly the spent "doctor" solution was run to waste, but recently the attention of refiners has been called to a new process whereby the spent plumbite is recovered. Approximately 95 per cent. of

the lead compounds in the spent sludge are recovered, 85 to 90 per cent. in the form of litharge and the balance as lead chloride, both of which are soluble in caustic soda solution and suitable for the preparation of "doctor."

The F.C. Process has been in commercial operation for some time, and all the spent "doctor" sludge from the refinery of the American Refining Properties, at Wichita Falls, Texas, has been reclaimed over and over again at a very small cost with complete success. The recovered litharge is in a finely-divided state and very soluble in caustic soda solution, so that it is unnecessary to have heating coils in the "doctor" mixing tanks. The sulphur is recovered as elemental sulphur and is very soluble in the treating mixture that is used to "break out" the mercaptides resulting from the plumbite treatment. The "doctor" solution containing the precipitated sulphur reduces the treating time by about one-half.

The striking thing about the "doctor" solution made from recovered litharge is that it is superior to "doctor" made from fresh chemicals and costs less than half as much as the fresh materials. The object of the F.C. Process is to reclaim all the chemicals from the spent sludge. The caustic soda and sulphur are not used up but are recovered along with the litharge.

The operations involved in the F.C. Process are collecting the spent sludge from the agitators by placing an ordinary steel tank below the level of the draw-off cocks of the agitators, separating the constituents of the sludge into a top layer of oil, an intermediate layer of caustic soda solution, and a lowermost layer of lead sulphide. The gasoline is drawn off to rerun, the caustic soda is run down to be used for the preparation of "doctor" in subsequent operations, and the solids which remain are washed to remove all the caustic soda. The lead sulphide sludge is then transferred to the conversion tank and mixed with hydrochloric acid from a previous batch. A stream of chlorine is introduced into the sludge and the contents of the tank agitated by a mechanical agitator until about $\frac{1}{4}$ lb. of chlorine is present for every 1 lb. of lead sulphide. The time of conversion is usually about 3 hours for 1000 lbs. of sulphide, but it is possible to speed up the conversion. After the conversion the contents of the tank consist of litharge, sulphur, and a small proportion of lead chloride whilst the liquid will be a dilute solution of hydrochloric acid. After settling, the contents of the tank separate into three layers, the upper is a mass of flocculent elemental sulphur, the middle layer a dilute solution of hydrochloric acid and the bottom layer a mixture of finely divided litharge, some sulphur, and traces of lead chloride. After being freed from HCl, the litharge admixed with sulphur is ready to be used in making "doctor" solution, and the caustic solution which had previously been recovered in the settling operation is used for this purpose. There is usually enough sulphur entrained in the litharge to complete the reaction of the "doctor" solution with the oil under treatment. Thus practically all the chemicals are restored to their original usefulness and may be reclaimed again and again.

The chemistry of the process may be stated thus:—

F.S. Process.

$$H_2O+Cl=HOCl+HCl$$
$$HOCl+PbS=PbO+S+HCl$$
$$PbO+2NaOH=Na_2PbO_2+H_2O$$
$$2RSH+Na_2PbO_2=(RS)_2Pb+2NaOH$$
$$(RS)_2Pb+S=PbS+2RS$$
$$Na_2PbO_2+H2S=PbS+2NaOH.$$

R. P.

823. Adhesion Tension Values of Different Types of Carbon Black against Water and against Benzene. F. E. Bartell and C. N. Smith. *Ind. and Eng. Chem.*, 1929, **21**, 1102.

The method used depends on the measurement of the pressure set up when a given liquid displaces air or a second liquid from a membrane of finely divided solid material packed in a displacement cell. High displacement pressures were obtained with nearly all of the carbon systems studied. Compressed nitrogen was used to secure a pressure balance, and the pressures were measured by a standard gas-pressure gauge graduated in kilograms per square centimetre. Adhesion tension values were determined for seven different types of carbon black against water and against benzene. The carbons were subjected to heat treatment and evacuation. It was found that reliable data could be obtained only when the carbon particles were so large that compressed membranes of them had pore radii greater than (approximately) 2×10^{-6} cms. It was shown that different commercial carbon blacks possess different adhesion tension values against water and against benzene, and that heat treatment alters the adhesion tension of these carbons against different liquids. W. L.

824. Process and Apparatus for the Purification of the Gaseous Cloud Formed by the Heat Transformation of Heavy Oils. C. Chilousky. E.P. 302,292. Oct. 17, 1929. Appl. Dec. 13, 1928. Conv. (Fr.) Dec. 13, 1927.

In this process the gaseous cloud is circulated upwardly through water-cooled walls formed by a large number of vertical tubes of small cross-section spaced slightly apart. The temperature of the cooling water is maintained between 40° and 60° F. The cloud may be cooled by passing through a water-cooled, double-jacketed metal cooler before being admitted to the purifying condenser, or by being mixed with gases which have been previously cooled and treated. W. L.

825. Comparison of Acetylene Black with Gas Black and Lampblack. T. R. Dawson and N. H. Hartshorne. *Trans. Inst. of Rubber Industry*, 1929, **5**, (1), 48.

In the files of British patents from 1830 onwards may be found references to all qualities of carbon black in rubber, for use as a pigment, as a reinforcing and toughening agent; in high-pressure packings and oil-resisting rubbers, as a protection against the effects of light and to render rubber a conductor of electricity. The three types of carbon black discussed in this paper are of the strongly reinforcing type: gas black, obtained by deposition on metal surfaces from the flames of burning gas; lampblack, obtained by incomplete combustion of organic matter rich in carbon, such as oils, fats, greases, tars, resins, etc.; and acetylene black, obtained by cracking processes.

In dealing with the chemical examination of carbon black, the table of results given shows that the acetylene black is the most pure, while the lampblacks are definitely the least pure. Although in general the amounts of impurity are so slight as to appear negligible, it may be noted that these impurities must be distributed in an ultra finely divided state in the minute black particles. They may therefore be capable of exceptional surface or catalytic effects. This point is mentioned later in discussing the effects of the blacks on organic accelerators.

With regard to physical examination, it is shown that the colour varies with different samples of each type of black, while the pigment power is the greatest in the case of gas black and least in the case of the acetylene black.

The differences, however, are of no practical significance in the rubber industry. The measurements of bulk are highest with lampblack and lowest with gas carbon, but the results only have a practical interest in regard to transport, storage and general handling. The acetylene black has the highest specific gravity, while the gas carbon and lampblack are about the same. The particle size is greatest in the case of the gas carbon and least with lampblack.

Each of the blacks was mixed in five proportions, by weight with 95-rubber-5 sulphur stock. The mixings were press cured over ranges of time and the optimum cures selected by means of the usual tensile tests. An examination of the mechanical properties of the mixings shows that, excluding the tensile strength and reinforcing power, in which the gas and acetylene blacks are almost identical, the gas black is always intermediate in properties to the other two. In properties associated with strength, such as rigidity, energy absorption and hardness, the gas and acetylene blacks are closer together and the lampblack stands somewhat apart. In properties associated with elasticity, such as permanent set and resilience, the gas black is nearer to the lampblack and the acetylene black stands apart. The magnitude of the differences indicate the variable nature of carbon black in its different types, and it is obvious that each type has specific advantages.

In experiments with "technical mixings," evidence is given of the persistence of the characteristics of the blacks, for the order of magnitude of the differences is the same for both complex and simple mixings. Permanent set stands out as the most sensitive test of the series.

Accelerated mixings containing organic accelerators show on examination that the gas black falls between the other two in the majority of cases, and is nearer to the acetylene black than to the lampblack. This is true in spite of the marked differences created by the different accelerators with any one black.

The cause of the variability in different batches of a single black probably lies in the slight differences in surface condition of the powders which modify the amount of "grip" between rubber and black and the state of flocculation of the black in rubber. The variability emphasises the importance of selection of black to suit the work in hand and also the fact that the indiscriminate use of one black for all purposes will in many cases result in inferior products. W. H. T.

826. A Note on Gas Black for Rubber Manufacture. The Effects and Detection of Grit. G. Gallie. *Trans. Inst. of Rubber Industry*, 1925 (**1**), 247.

The object of the paper is to draw attention to the type of variability in commercial samples of gas black and to discuss, briefly, the tests used to detect grit and the possible ill-effects of this impurity when used in rubber compounding. The bulk of the gas black on the market is made by the Channel process, in which burning natural gas is allowed to impinge on the metal surfaces of steel channels. The distortion of the steel channels results in the failure of the scrapers to make contact with the entire sooty surface, with the consequence that portions of the black are not removed, but pass and repass through the flames. Thus, alteration in physical properties takes place, and the black is burnt into hard gritty flakes. Portions of this altered material become detached from the channels and contaminate the bulk of the product. None of the processes used for the subsequent removal of this grit appears to be continuously successful. The fragments of grit are variable in size, show a greyish-black colour and are occasionally rust marked, some surfaces having a sub-metallic lustre. When washed, the particles are clean to handle and are hard enough to resist crushing with the fingers. The grit on

incineration invariably leaves a higher proportion of ash than the normal product contains, appreciably more matter soluble in acetone or petroleum ether, but has the same specific gravity as the normal black.

The occurrence of grit in gas black is generally investigated by sieving, the removal of the finely divided material being assisted by carrying out the process under a stream of water. Forty-five samples of carbon black were examined, and not one was found to pass completely through a 300-mesh sieve. Tests were also made with an 80-mesh and 0·6655 per cent. and left on the 300-mesh sieve—*i.e.*, more than seven times as much. The grit is distributed very irregularly not only throughout a consignment but throughout the individual bags. The possibility of grit being present in a delivery is by no means precluded by a negative result on samples drawn from one or two bags.

The author has devised an apparatus (E.P. Appl. 24,987, Oct. 7, 1925; G. Gallie and B. D. Porritt) which makes it possible to carry out tests for the presence of grit in gas black under readily reproducible conditions. As a result, one of the principal difficulties in the way of standardising the testing of gas black has been removed, and it only remains for suppliers and consumers to agree on methods of sampling and standards of quality.

The presence of grit in the gas black used for rubber manufacture gives rise to trouble in the following directions: (*a*) degradation of the tensile and other mechanical properties; (*b*) discontinuity in rubber films required to retain or exclude gases or liquids; (*c*) unsatisfactory surface finish; (*d*) points of weakness in rubber articles subjected to severe mechanical usage or repeated compression and stressing. W. H. T.

827. Compositions for Road Making. Hewitt Ltd. and A. Hewitt. E.P. 319,904. Oct. 3, 1929. Appl. Sept. 4, 1928.

The patent describes an improved composition for the treatment of aggregate in road making and for the renewal of existing road surfaces. The composition is made by mixing 5 lbs. of rubber with 10 gallons of bituminous oil and heating the mixture to the boiling point of the oil. 1 lb. of powdered resin is added to the hot material, and after cooling 1½ lbs. of crushed oil shale is also added. The final product is employed for the treatment of a suitable mineral aggregate such as slag, granite or gravel dust. Proportions of Portland cement and calcium chloride are added to the mixture as required. W. H. T.

828. Improvements in the Manufacture and Production of Plastic and Elastic Polymerisation Products of Diolefines. I. G. Farbenindustrie. E.P. 292,103. Aug. 29, 1929. Appl. June 8, 1928.

This invention provides a process whereby the polymerisation of diolefines to produce plastic and elastic polymerisation products, is greatly accelerated by emulsification in suitable aqueous liquids in the presence of hydrogen peroxide. H. E. T.

829. An Improved Cold Asphalt and Application thereof. O. Y. Imroy, assr. to I. G. Farbenind. Akt. E.P. 317,496, Aug. 16, 1929. Appl. March 16, 1928.

It is claimed that the cold asphalt made in accordance with this invention yields a road surface essentially more durable than one afforded by the emulsions, or a surface of equal durability, with consumption of less material. The invention is based on the observation that humic acid insoluble in water determines the formation from bitumen and water of a coarse dispersion which differs somewhat in physical properties from the current bitumen emulsions, and can hardly be regarded as an emulsion in the ordinary sense. The use of colloid mills, steam blasts, and other strongly dispersing agencies are not needed. W. H. T.

830. Manufacture of Calcium Hypochlorite. L. Mellersh-Jackson, from Mathieson Alkali Works. E.P. 316,447, Aug. 1, 1929. Appl. Aug. 18, 1928.

Calcium chloride is eliminated from calcium hypochlorite products by treatment with an alkali hypochlorite; *e.g.*, sodium hypochlorite, whereby calcium hypochlorite and alkali chloride are formed. The process is applicable to dry calcium hypochlorite products, to slurries or to filter or press cakes. During treatment sufficient water must be present to permit thorough agitation and mixture. Following the treatment the product may be filtered and dried or dried directly, with or without pressing. The alkali hypochlorite may be used as such or in solution. For example, a solution of 55 lb. of caustic soda in 65·3 lb. of water may be chlorinated until not more than about 6 per cent. of the alkali remains unchlorinated. It is then advantageous to remove precipitated salt. The solution may then contain about 29 per cent. of sodium hypochlorite, 7·5 per cent. of sodium chloride and 1·5 per cent. of sodium hydroxide. This solution may then be used or, if desired, sodium hypochlorite pentahydrate may be frozen out, separated and used.

Thus, 47lb. of sodium hypochlorite and 73lb. of water are added to 500lb. of a filter cake containing about 50·1 per cent. of calcium hypochlorite, 6·9 per cent. of calcium chloride, 5·7 per cent. of calcium hydroxide, 0·3 per cent. of sodium chloride and 37 per cent. of water, and the mixture is thoroughly stirred and dried without pressing. The product contains less than 0·5 per cent. of calcium chloride. W. S. N.

831. Manufacture of Calcium Hypochlorite. L. Mellersh-Jackson, from Mathieson Alkali Works. E.P. 317,233, Aug. 15, 1929. Appl. Aug. 18, 1928.

Calcium hypochlorite substantially free from calcium chloride is prepared by chlorinating an aqueous mixture of lime and a caustic alkali (*e.g.*, caustic soda), the alkali being in excess of that chemically equivalent to the lime, treating the chlorinated mixture with a calcium compound (*e.g.*, calcium chloride) to convert alkali hypochlorite to calcium hypochlorite, and separating calcium hypochlorite from the product by filtration or evaporation.

Thus, a mixture of caustic soda (120lb.), high grade lime (95 per cent. of $Ca(OH)_2$) (78lb.) and water (562lb.) is chlorinated (*e.g.*, at about 20°) until only about 2 per cent. of free alkali remains. If the type of agitation employed makes it necessary, more water may be added. Finely-divided calcium chloride ($CaCl_2.2H_2O$) (36lb.) is then added with stirring. Salt and calcium hypochlorite are then precipitated. The resulting slurry may then be taken to dryness to give a product containing about 47 per cent. of calcium hypochlorite, the balance being principally sodium chloride. Alternatively, the calcium hypochlorite may be removed from the slurry by filtration and dried either with or without pressing, thus yielding a product of higher calcium hypochlorite content. The proportion of sodium chloride may be further reduced by adding water prior to filtration of the precipitated calcium hypochlorite. W. S. N.

832. Improvements in the Manufacture and Production of Hydrocarbons of High Molecular Weight. J. Y. Johnson. E.P. 298,584, Oct. 9, 1928. Appl. June 9, 1927.

A process is described for the manufacture and production of hydrocarbons of high molecular weight, and in particular, products of the nature of caoutchouc. The method consists in polymerising the products containing substantial quantities of hydrocarbons containing at least four carbon atoms and having at least two conjugated double bonds, which are obtained by the

destructive hydrogenation of carbonaceous materials. If the initial material does not contain a sufficient amount of unsaturated hydrocarbons, but contains substantial quantities of saturated hydrocarbons such as butanes, pentanes, hexanes, cyclopentane or cyclohexane, it is subjected to a preliminary dehydrogenating treatment. Whether or not this preliminary treatment is necessary, is determined by shaking with sulphuric acid, when, if large quantities of unsaturated compounds are present, considerable darkening of the material will occur. Examples illustrating the scope and manner in which the invention is carried out are quoted. H. E. T.

833. Manufacture and Production of Carbon. J. Y. Johnson, from I. G. Farbenind. E.P. 319,140, Sept. 19, 1929. Appl. Sept. 17, 1928.

The invention is an improvement on E.P. 286,845, according to which carbon is produced by heating CO in the presence, for example, of small mounts of iron carbonyl vapour. The present invention claims advantageous results as regards yield, purity, and quality of the products by the addition of small quantities of water vapour (0·5—5·0 per cent. by vol.) to the carbon monoxide, which may with advantage be mixed with CO_2, N_2, H_2, or gaseous hydrocarbons. 0·1 to 0·5 per cent. by vol. of a volatile metal compound which decomposes CO, for example, the carbonyl of iron, nickel, or cobalt, or an organic compound such as the acetyl acetonate of a metal of the iron group is added, and the whole is passed downwards through a vertical heating tube. Temperatures employed may vary from 350–700° C., and the pressure may range from below atmospheric to over 200 atmospheres. The carbon black may be removed by worm conveyor from a settling chamber at the foot of the tube. By virtue of its high state of dispersion the carbon black so produced is eminently suitable for use in printing inks, and in the rubber industry.

As an example, CO containing about 0·18 per cent. if iron carbonyl and about 0·8 per cent. of water vapour was passed at atmospheric pressure through a tube heated to 500° C.; 60 per cent. of CO was decomposed into carbon and CO_2, which latter constituted 45 per cent. of the effluent gases. L. H. A.

834. Gilsonite and Related Bitumens (Contd.). H. M. Langton. *Ind. Chem.*, 1929, *5*, 383.

Gilsonite is graded commercially in two ways, first into screened and unscreened, and secondly into "Selects" and "Run-of-Mine" (or simply Gilsonite). The intention of the second type of grading is to differentiate between the softer ore and the harder, more weathered, and less readily fusible ore. "Selects" is dug from only those portions of the mine where workings have proved the ore to be uniformly of the softer grade. The extremely hard grades sometimes cause "livering" in paints, and also require a greater amount of drying oil, or solvent, to bring them to the desired consistency than do the softer grades.

Gilsonite has jet black colour, conchoidal fracture, bright lustre, red-brown streak, sp. gr. 1·01 to 1·10, a hardness of 2 and a melting point of 250° to 350° F. Ash is usually between 0·15 and 0·75 per cent., and is mainly insoluble silicates, ferric oxide, and traces of nickel oxide. Total bitumen soluble in carbon disulphide may be as high as 99·5 per cent. Abraham gives the fixed carbon as 10 to 20 per cent., and a distillation test gave

0–150° C.	9·34	per cent.	distillate.
150–200° C.	5·35	,,	,,
200–250° C.	12·84	,,	,,
250–300° C.	28·99	,,	,,

Above 300° C. the material cokes.

Gilsonite is insoluble in ethyl, propyl and amyl alcohols, but soluble in all proportions in ethyl ether and carbon disulphide. It is freely soluble in the usual paint solvents. It is used principally for the manufacture of paints, varnishes, and japans, also in electrical insulation work and ink manufacture. Its uses in these industries are outlined. It is also used to some extent in prepared roofing and flooring materials, and in the rubber industry.

Glance pitch somewhat resembles Gilsonite, but has a higher sp. gr. and a black streak. Its sp. gr. is 1·10 to 1·15. It is claimed that no other native bitumen equals it in respect to lustre, melting point, colour intensity and elasticity. Grahamite is a black asphaltite with a lustre varying from dull to very bright, a conchoidal to hackly fracture, black streak, sp. gr. of 1·15 to 1·50, a hardness of 2 to 3, and a melting point of 350° to 600° F. As much as 50 per cent. mineral matter may be found in association with it. Grahamites do not really melt, but intumesce and swell at high temperatures, this differentiating them from all other bitumens except albertite. Its chief use is in the manufacture of prepared roofing. When fluxed with a heavy asphaltic flux it is less susceptible to heat than Gilsonite and is more rubbery and elastic.

Manjak is a term loosely applied to glance pitch from Barbados and to a soft variety of Grahamite from Trinidad. Barbados manjak is black, with a black streak, bright lustre, hardness of 2, conchoidal fracture, sp. gr. about 1·10, and melting point 320° to 430° F. It contains about 90 per cent. of carbon, of which 25 to 30 per cent. is fixed carbon and has a solubility of 97 to 99 per cent. in carbon disulphide.

Trinidad manjak has about the same properties, except that its which is 1·17 to 1·175, its melting point 350° to 438° F., carbon 84 per cent. of sp. gr. 3·15 to 3·50 per cent. is fixed carbon.

Manjak is primarily used for the manufacture of paints and varnishes, particularly when mixed with mineral oils. Another important use is as a pipe cement used in rotary drilling for oil. W. L.

835. Manufacture of Calcium Hypochlorite. Mathieson Alkali Works. E.P. 320,182, Oct. 10, 1929. Appl. Aug. 18, 1923.

An improved process of making hypochlorite is described, which comprises chlorinating a lime slurry and salting out calcium hypochlorite from the chlorinated mixture by the addition of sodium chloride. The salting out operation is carried out with the addition of a diluent solution substantially saturated with calcium hypochlorite (*i.e.*, the solution remaining after separation of the calcium hypochlorite). The lime slurry should contain between 24 and 37 pounds of water per 10 pounds of lime. W. H. T.

836. Manufacture of Bleaching Powder. J. W. Moore, A. Lamble, and Imperial Chemical Industries Ltd. E.P. 317,572, Aug. 22, 1929. Appl. June 9, 1928.

High-strength stabilised bleaching powder is produced by combining in one operation the manufacture and all or a large part of the drying of the material by utilising the heat of reaction, and mechanically agitating the material so that the moisture is removed by the warm gas during the manufacture. Preferably a large controlled volume of dilute chlorine is circulated through the apparatus, moisture being removed from the effluent gas before it is recirculated. For example, a Moore rotary tube (E.P., 113,457) may be used. This consists of a long iron cylinder lined with glazed earthenware, supported at an inclination of about 1 in 24 on roller bearings and caused to rotate by worm gearing. Lime is fed into the upper end, and bleaching powder harged at the lower end. The hot exit gas is suitably withdrawn from

the lime feed end and is passed through earthenware atmospheric coolers. If necessary, further cooling is obtained by the use of brine. The condensed water is drained away and the dried exit gas is brought up to strength with new chlorine and, if desired after preheating, is delivered to the inlet pipe of the same or a similar reaction tube. In its passage through the tube the gas is warmed to about 60°. Satisfactory dehydration is obtained if, say, 10,000–12,000 cubic feet per hour of weak chlorine are circulated through a tube producing about 32 tons per week of high-strength bleaching powder, *e.g.*, containing about 37 per cent. of available chlorine. W. S. N.

837. Some Observations on Carbon Black. C. M. Carson and L. B. Sebrell. *Ind. and Eng. Chem.*, 1929, 21, 911.

Different carbon blacks have certain definite effects upon the vulcanized rubber stocks with which they are mixed. The experiments described in this article may be grouped under four main heads. (1) adsorption, (2) effect of heat, (3) reaction with sulphur and zinc oxide, (4) dispersion or rate of settling. In studying adsorption the benzoic acid method was first used, but later the use of iodine solutions was adopted. The effects of time, temperature, concentration of solutions, weight of sample, presence of inorganic salts and the pH of water used all have to be taken into account. Standard procedure was established and followed in all cases. The blacks used in the experiments were two channel blacks, two grades of thermatomic, one varnish black, and three high-temperature blacks.

The adsorptive capacity of the black is a measure of the rate of cure of the rubber mix, low adsorptive blacks giving a faster curing stock than high adsorptive blacks. Blacks made at high temperatures (1000–1200° C.) are ordinarily better stiffening agents than channel black. On some blacks the effect of temperatures from 500° to 1200° C. is to render them highly adsorptive, and also to give a rubber mix which cures faster and has a higher modulus. This appears to be a contradiction of the previous statement, that high adsorption means slow cure, and the writers have no explanation for this behaviour. The reaction of carbon blacks with sulphur and zinc oxide in boiling xylene liberates a substance having accelerating properties in a pure gum mix, so that while the addition of black to a rubber-sulphur mix slows down the time of cure, the addition of black to a rubber-sulphur-zinc oxide mix accelerates the curing. An indication of the stiffening action of a carbon black may be secured by measuring the amount of carbon, dispersed in a thin rubber cement, which cannot be centrifuged out in a given time. The union of black and rubber is a powerful force, as is shown by the fact that high quality blacks cannot be totally centrifuged from a rubber cement even at fairly large dilution, and this is true of no other pigment.

W. L.

838. Regeneration of Alkaline Purifying Beds. R. C. Wheeler and P. W. Prutzman. U.S.P. 1,728,156, Sept. 10, 1929. Appl. June 24, 1927.

When petroleum vapours containing phenols or naphthenic acids are passed through permeable beds of solid alkalis, soaps and salts are formed on the surface of the alkalis. Under certain conditions these bodies drain away, exposing a fresh surface for the continuance of the reaction. Under other conditions, notably when the vapour velocities are low, the products formed adhere to the surface of the alkali, inhibiting further reaction.

If, however, a small quantity of water vapour be mixed with the oil vapours prior to their entry into the purifying bed, the reaction product may be detached as fast as it is formed, and the alkali maintained in an active condition until it is consumed.

A quantity of water ranging from $\frac{1}{10}$ per cent. to 1 per cent. of the volume of oil distilled will usually provide a sufficient amount of water vapour for the purpose. An alkali bed which has become coated with soaps may be rendered reactive by temporarily admitting a larger quantity of steam than the above, but any excess merely dissolves the alkali without serving any useful purpose.

H. B. T.

Analysis and Testing.

839. Specific Gravity of Asphalt by Means of a Hydrometer. H. R. Berry, E. H. Smith and F. R. Lang. *Ind. Eng. Chem.* (Analytical Ed.), 1929, 1 (3), 164.

The asphalt samples are contained in cylindrical sheet-iron vessels 3·18cm. diam. and 15·24 cms. deep, immersed in a constant-temperature oil bath. This bath must maintain its temperature to plus or minus 1° F. during the determination, which is carried out between 290 and 300° F. The asphalt samples are heated to slightly above the bath temperature and then poured into the containers. When the temperature, as indicated by a thermometer placed directly in the asphalt, becomes constant the hydrometer is floated and the gravity and temperature recorded. A 10-16 cm. A.P.I. scale hydrometer has been found convenient and satisfactory. Tables are given for the conversion of the readings obtained to specific gravity at 60° F. The method has been checked from time to time by means of a pyknometer and has been found to give uniformly satisfactory results.

W. L.

840. Quantitative Determinations of Mercaptans in Naphtha. P. Borgstrom and E. Emmett Reid. *Ind. and Eng. Chem.* (Anal. Ed). 1929, Vol. I., 4, 186.

A method is outlined for the estimation of mercaptans in petroleum distillates, based on titration with silver nitrate and ammonium thiocyanate. Alternatively the sulphur can be removed by silver nitrate and the remaining sulphur in the naphtha can then be determined by the lamp method. Acidified cadmium chloride did not remove the mercaptans studied. Mercury used for the removal of elementary sulphur as first recommended by Ormandy and Craven and later altered by Faragher, Morrell and Monroe does not remove mercaptans.

Glacial acetic acid and zinc used for the reduction of disulphides cause a partial decomposition of the mercaptans, and thus the titration method cannot be employed. The percentage of sulphur must be determined by some method, such as the lamp method, applied before the reduction and after the removal of the resulting mercaptans and other substances formed from the disulphides.

The mercaptans studied in this work were ethyl, n-propyl, isopropyl, n-butyl, isobutyl, secondary butyl, n-amyl, isoamyl, feota-theol-2 and benzyl.

H. E. T.

841. Methods of Testing Bitumen. H. Bösenberg. *Petroleum*, 1929, 25 (1275-1279).

The determination of the dropping point, solidification, and the stability value of a bitumen is described in this article. The dropping point is the temperature at which a coating of bitumen on the bulb of a thermometer drops under its own weight. Two glass shells with outlets 3 mms. wide are

filled with bitumen, placed on glass plates in a hot-air bath, and attached while still soft to the thermometer bulb. The thermometer is cooled to room temperature, inserted through a cork into a test-tube with some sand at the bottom, and heated in a 3-litre beaker of water so that the temperature rises 1° C./minute. The temperature at which there is a distinct curvature at the bottom of the glass shell denotes the commencement of fluidity which is usually in the same region as Kraemer-Sarnow's melting point. The dropping point is that temperature at which the first drop falls and is about 20° C. higher than the melting point. Bitumina of high-melting points are heated in a bath of paraffin oil instead of water. The length of the thread of asphalt at the moment of dropping is determined by measuring in cms. on the glass, by means of a wax pencil, the distance of the drop from the thermometer orifice.

For solidification point determinations the thermometer bulb is covered with a layer of asphalt 1–1·5 mms. thick, cooled to room temperature in an air bath, and the temperature observed at which it is no longer possible to scratch the pitch with a finger-nail or to produce imprints on it.

The international stability test, A.S.T.M., D6-27, is the determination of the loss in weight by evaporation after five hours' heating at 163° C. 50 gms. of dry bitumen are added to a cylindrical metal container of known weight, and rotated at the rate of 5–6 revolutions/minute in a specially constructed oven fitted with a perforated plate containing divisions 25 cms. in diam., 50 gms. of the same bitumen are placed in another tarred container and used as a standard, and the whole of the thermometer bulb is immersed. The oven is closed and after five hours' treatment, the samples are cooled and weighed. The results of loss in weight by evaporation should be within $\pm 0{\cdot}5$ per cent. of the standard when the loss by evaporation does not exceed 5 per cent.; for higher values a deviation of 0·01 per cent. is allowed for each 5 per cent. loss by evaporation. If the softening point and penetration value change after heating, a fresh sample must be prepared as the standard. The Central Institution for Asphalt and Tar Research use 25 gms. asphalt in a glass Petri dish of 64 sq. cms. surface, and a bituminous layer of only 0·375 cms. depth as opposed to 2 cms. in the A.S.T.M. method.

The physical properties of their pitches as well as those of the American Asphalt Association are given, also of mixtures of these. It has been found possible by mixing given grades of bitumina to obtain any desired degree of penetration value and melting point. The article is supplemented with numerical data.

W. S. C.

842. Determination of Total Moisture in Carbon Blacks. C. M. Carson. *Ind. Eng. Chem.* (Anal. Ed.). 1929, I., **4**, 225.

The total moisture in carbon blacks has been determined by an adaptation of an old method, similar to that used by Allen and Jacobs for the determination of water in tar.

Five gms. of carbon black were placed in a 500 cub. cm. round-bottomed flask, with 25–35 cub. cm. of dry xylene and 200 cub. cm. of dry mineral oil. A short air condenser led to the bottom of a 25 cub. cm. distilling flask, which was connected to two or more calcium chloride tubes. The flask containing the sample was heated to 150–175° C. in an oil bath, a stream of dry nitrogen being passed through the apparatus. The water and xylene were distilled into the small distilling flask, and thence, by warming in a water bath, into the calcium chloride tubes, the current of nitrogen being continued. The increase in weight of the calcium chloride tubes gave the percentage of water in the carbon black under examination.

H. E. T.

843. A Process and Apparatus for the Volumetric Analysis of Gas, such, for instance as Firedamp in the Atmosphere. J. G. Daloz. E.P. 319,177. Sept. 19, 1929. Appl., Jan. 15, 1929.

This invention relates to processes and apparatus for the volumetric analysis of gas, and consists in improvements or modifications in the method and apparatus described in the co-pending Patent application No. 300,281. In the present invention the common distributor utilised in the parent application is replaced by two distributors having a single control, and means provided for isolating the combustion chambers from the measuring system while combustion takes place. H. E. T.

844. Analytical Reactions of Tetraethyl Lead. G. Edgar and G. Calingaert. *Ind. Eng. Chem.* (Anal. Ed.), 1929, I., **4**, 221.

In this paper the authors describe methods whereby tetraethyl lead and related alkyl lead compounds either in concentrated solutions or in gasolines can ready be determined. The limits of accuracy of the methods and their applicability under various conditions is also discussed. H. E. T.

845. Determination of Sulphur in Petroleum Oils. R. C. Griffin. *Ind. and Eng. Chem.* Anal. Ed., 1, III., 167.

It is well known that some mercaptans and other organic sulphur bodies give low sulphur values when estimated either by bomb or lamp methods. In the bomb method this is due to oxidation to sulphonic acids and similar bodies, the barium salts of which are water soluble. The sulphur present as sulphonic acid may, however, be recovered by refluxing the filtrate from the barium sulphate with concentrated hydrochloric acid which converts the sulphonic acid to sulphuric acid. A. R. S.

846. The Determination of Paraffin Hydrocarbons in Motor Fuels by Sulphonation. G. Kuhn. *Chem. Ztg.* 1929, 72 (701-702).

Previous work on this subject by Heilingötter, Pritzker and Jungkunz has produced no satisfactory method for the determination of 0–100 per cent. benzene content in benzene-benzol mixtures. Kuhn, utilised one part fuel to two parts acid as in Pritzker and Jungkunz's method, and fuming sulphuric acid containing 4·8 per cent. free SO_3. The sulphonation was carried out in a 150 ccs. mounted glass flash with a fused burette 60 cms. long and reading to $\frac{1}{5}$ of a c.cm. The opening of the burette could be closed by means of a ground glass stopper. The initial substance consisted of chemically pure benzol (specific gravity 0·8833 at 15° C.), and normal benzene (specific gravity 0·7005 at 15° C.). 10 c.cms. benzol-benzene and 20 c.cms. fuming H_2SO_4 were used in each experiment.

Kuhn's results for benzene-benzol mixtures of 0–30 per cent. benzene content are greater than those of Pritzker and Jungkunz. The differences between the two results increase after 30 per cent. rise to a maximum at 50 per cent. benzene content, and then gradually decrease with higher benzene content. A curve is given showing the relationship of the benzene content of benzol-benzene mixtures to the sulphonation value. It is possible to estimate the benzene content from the sulphonation residues of fuels of any composition. The accuracy amounts to about 2 per cent., but this can be increased by using greater quantities of fuel and acid. W. S. C.

847. Rapid Method of Testing High Sulphur Gases in the Field. M. J. Leahy. *Refiner*, 1929, **8** (10) 79.

The necessary apparatus is a flue-gas analyser similar to the Orsat-Muencke, specific gravity bottle, stop watch, dry test gas meter, set of gas washers

preferably made from two-inch pipe connected in series with quarter-inch pipe, a pitot tube and manometer, a number of charcoal tubes filled with activated charcoal, and the necessary solutions. The first pipette of the gas analysis apparatus is filled with a strong aqueous solution of iodine in potassium iodide, the second with strong potassium hydroxide solution and the third is left empty or filled with pyrogallol if the presence of air is suspected. The gas washers are partly filled with strong potassium hydroxide solution. The liquid in the levelling bottle of the analyser is a 10 per cent. solution of salt in water.

The sample is taken, with the usual precautions, from the drain cock of the oil and gas separator the pressure on the separator being noted. The hydrogen sulphide content is obtained by absorption in the iodine and then the carbon dioxide by the potassium hydroxide.

The train of gas washers is then connected to the drain cock and the specific gravity of the gas issuing from it determined by the specific gravity bottle, the outflow time of the gas from the bottle compared with that of air giving the necessary data. From this the number of cubic feet of gas necessary to saturate the charcoal with gasoline can be calculated. Connect the test meter to the gas washers and after regulation pass the requisite quantity of issuing gas into the charcoal tube. This tube is closed and sent to the control laboratory for distillation for gasoline content. The volume of gas issuing from the well is determined by the pitot tube and manometer. As the gas passing the test meter is free of hydrogen sulphide and carbon dioxide, corrections must be made for the loss in volume due to these constituents if the amount of gasoline in the untreated gas is required. W. L.

848. The Determination of the Tar, Number of Transformer and Switch Oils. E. Locher. *Petr. Zeit.* 1929, **25**, p. 1215.

An improved method for the determination of the tar number of transformer and switch oils is described, in which 50 gms. of the oil are placed in a 200 cc.s. Ehrlenmeyer flask with 10 c.cms. 7 per cent. caustic soda solution and heated in the absence of oxygen at 120° for 24 hrs. The oil is then treated by the ordinary precipitation method without separating the soda. The values of the tar numbers obtained in a series of tests with the present method and the ordinary precipitation method are quoted. The described method gives higher results, the maximum deviation being 0·03 per cent. higher than the ordinary precipitation method, and moreover it is quicker and cheaper.

W. S. C.

849. Fractional Distillation Analysis. W. J. Podbielniak. *Oil and Gas J.*, 1929, **28** (21), 161.

The general precautions which should be observed in using any precise method of analysis for gas and gasoline are described. The apparatus used is that described in Abs. No. 342. The important variables of the apparatus and the method of analysis are discussed in detail, to show their effect on the sensitivity and accuracy of the apparatus and how to control them to obtain the utmost practical accuracy.

The most important variables affecting fractionation in this apparatus are the adjustment of the column packing, the rate of distillation, which affects the reflux ratio, and the hold up of liquid on the column packing.

It should be possible by observing the proper precautions and operating the apparatus fairly efficiently according to the principles explained to secure an accuracy at least within one-tenth of one per cent. for individual percentages.

Various data are presented showing typical analyses and checks obtained in practice. These checks show an accuracy from a few hundreths of one per cent. to two or three-tenths per cent. depending on the difficulty of separation of the compounds analysed and the carefulness of the operator. W. L.

850. The Quantitative Determination of Benzene and Toluene in Gases. Schultz. *Czecho. Chem. Comm.*, 1929, 1, 4.

It is shown that Gustavson's method of detecting aromatic hydrocarbons in gasoline by bromination in the presence of anhydrous aluminium bromide can be applied to gas analysis. The bromination products other than those of the aromatics can be washed out with methyl alcohol. The time of treatment is 30 minutes at a temperature not lower than 20° C. It is essential that sufficient aluminium bromide be used and that the bromine be spread frequently over the walls of the reaction vessel during treatment. W. H. T.

851. Rapid Method for determination of Phenols. J. A. Shaw. *Ind. & Eng. Chem.* 1, III, 118.

The method was evolved to measure the phenolic content of ammonia liquors. Alcohols, amines, aldehydes, oils, organic bases and metallic salts must be first removed as these materially interfere with the determination. 10 c.cs. of the samples, which should be diluted to contain approx. 1 gm. per litre of phenols, are introduced into a pyrex test tube which is connected to another tube of water and at the other end to a Lisbig condenser. The two tubes are immersed in a Shaw bath and air passed into the first tube in such a manner and rate that it is saturated with steam when passing through the phenol, which has previously been acidified with sulphuric acid in the second tube. Thus phenols and volatile organic compounds pass through the condenser into a receiver. The distillate is made alkaline with caustic soda and the distillation repeated causing separation of all volatile organic matter. The acidified solution is redistilled and the phenols pass over, and the solution is diluted to approximately 30 pts. per million of phenol. Bromine water is added to the test sample and two others of known concentration is 30 and 35 ppm., the three are shaken at 18-20° C. and compared for turbidity due to the colloidal precipitate of bromine and phenol. The method cannot be used without concentration of solutions where less than 30 ppm. of phenols are present. A. K.

Refinery Plant.

852. Method and Apparatus for Treating Hydrocarbon Compounds A. Balmer. E.P. 317,508. Aug. 12, 1929. Appl. May 11, 1928.

This invention provides a method whereby hydrocarbon compounds, particularly petroleum oils, are converted into compounds suitable for use as motor fuels, the latter having usually a lower molecular weight and boiling point. The method may be regarded as an improvement on a previous specification, No. 207,276. The essential characteristics of the method may be outlined in the following manner.

A gaseous heat carrier is stoved up and maintained thus until it is above the temperature of conversion, when it is admitted to the reaction zone. The vapour thus produced is separated from the unvaporised liquid, and mingled with the heated gaseous carrier in the reaction zone in order to bring about a conversion of the vapour entirely in the vapour phase. The temperature of the reacting compounds is controlled in such a manner that the conversion

takes place under definite conditions of temperature and pressure to produce a composition suitable as motor fuel. The gaseous carrier is of such a quantity and has such a heating power, that it is able to furnish the entire heat necessary for the conversion. The apparatus for carrying out the method is fully described, and diagrams are provided. H. E. T.

853. Progress Made in Temperature Control (in Fractionating Towers). W. C. Begeebing. *Oil and Gas J.*, 1929, **28** (20), 165, 331.

For the automatic regulation of tower temperatures, throttling or floating control is the desideratum. The air-operated controller with diaphragm-meter valve is the type most widely used. The essential parts of this controller are the tube system with its sensitive bulb, the controller with its pilot air valve, and the diaphragm-meter valve. The mercury-actuated instrument is highly accurate and sensitive, and can cover a wide range of temperatures. The present tendency is towards the recording or indicating type.

The meaning of "throttling" depends on its application. A controller is throttling when it maintains the air pressure between 0 and 15 lbs. A diaphragm-meter valve is throttling when it is neither fully open nor tight shut. The specifying of the proper valve is as important as selecting the best type of controller. The best type of valve is that having V-port construction, as this gives a finer gradation of the valve opening and will utilise every increment of controlled air-pressure. It is also important to know the pressure of the reflux to be handled. G. S.

854. Prevention of Propagation of Flame in Conduits for Combustible Gases. T. R. Cave-Brown-Cave and H. Wood. E.P. 317,926. Aug. 29, 1929. Appl. June 1, 1928.

The flame arrester consists of a pack of annular metal discs, separated by narrow radial spacers, or of discs constructed with radial recesses or projections. The pack is held by flanges across an induction pipe, and the combustible gas is introduced through the edges of the pack of discs. If necessary the apparatus may be cooled by a water jacket. T. A. S.

855. Gas-Producing Apparatus and Process. J. J. O'Connor. U.S.P. 1,733,621, 1,733,622, and 1,733,623. Oct. 29, 1929. Appl. (*a*) Oct. 12, 1921; (*b*) Apr. 14, 1923; (*c*) Apr. 8, 1929.

(*a*) This comprises duplicate generators and gas receiver, the former having a chamber for gas-yielding material and duplicate gas-conducting connections. One upper and one lower of the said connections of each chamber is common to the two chambers for communication between them, and the other upper and lower connections of each chamber lead to the receiver; a separate valve-control effects circulation from each chamber to the receiver connection in either direction.

(*b*) Comprises two masses of coal or like carbonaceous material in separate gas generators, of which one mass is heated to incandescence, steam passed through, and the resultant hot water gas passed directly through the second mass and thence to a gas outlet. The direction of flow of the steam is reversed periodically without altering the order in which the two masses are attacked thereby.

(*c*) Two masses of carbonaceous material are placed in separate chambers, one mass is subjected to partial combustion, and the gas obtained passed directly through the second mass without substantial change or decrease of its sensible heat, and thence to a gas outlet. The flow of gas through the first mass is reversed frequently. W. S. C.

856. APPARATUS FOR TREATING PETROLEUM OILS. A. D. David. U.S.P. 1,733,651. Oct. 29, 1929. Appl. Feb. 7, 1923.

The invention relates to improvements in cracking plant for the pressure distillation of petroleum oils, of the type comprising a heating coil situated within a furnace, a vaporising chamber, dephlegmator, condenser, and receivers.

The improvements consist in the method adopted for recirculating the reflux oil from the dephlegmator in order to treat the charging stock and reflux separately during passage through the heating zone, with the object of lessening the deposition of carbon, and facilitating the cracking of the more refractory reflux.

The heating tubes consist of an inner and outer tube. Fresh charging stock is pumped through the inner tube whilst reflux oil is fed into the outer tube. The double tube terminates in the transfer line to the expansion chamber in which the oils mix and evaporate. The reflux oil firstly transfers heat to the charging stock, then receives the excess heat from the furnace and transfers sufficient heat to the inner tube to raise the raw oil to conversion temperature. R. P.

857. IMPROVEMENTS IN AND RELATING TO APPARATUS FOR SEPARATING IMPURITIES FROM OILS. Deutsche Werft A.G. E.P. 308,752. Aug. 22, 1929. Appl. June 21, 1928.

An apparatus is described, which is adapted to the separation from oils of water and other impurities. The small drops of water in the oil are caused to become agglomerated into larger drops on a guiding wall, or walls, which are inclined to the direction of motion of the ascending oil and to run along the walls to a water collecting space. Mechanical impurities having a greater specific weight are caused to become separated by deflecting plates arranged horizontally, which may be either curved or inclined laterally. H. E. T.

858. APPARATUS FOR MAKING OIL GAS. A. E. Dickerman. U.S.P. 1,728,400. Sept. 17, 1929. Appl. Aug. 25, 1922.

The apparatus is suitable for the production of gas in private houses, but has been produced in much larger units. The unit consists of a vertical casing lagged with asbestos and provided with a gas burner, a retort comprising a flat circular chamber is situated above the burner and has openings in the top plate to accommodate a central feed pipe and a number of heating tubes grouped in three concentric circles, so staggered that the hot gases from the burner pass close to the pipes. These pipes are welded into an annular header at their upper ends. The central opening in the header serves as an escape for the flue gases. Through it the feed pipe projects, and is connected to the oil and water supply, which are regulated by sight feed needle valves. The annular header has a conduit to conduct the gas to a cooler which condenses unchanged oil and water. The flue gases may be passed through a water heater to utilise the waste heat. The retort burns about one-tenth of its own products. R. P.

859. COOLING AND DEGASSING TOWERS. Theodoor Van Iterson and Madeleine Kuypers. E.P. 320,505. Oct. 17, 1929. Appl. Sept. 20, 1928.

By fitting fans, driven by electromotors, in cooling and degassing towers, of the type set out in patent No. 108,863, a forced draught is created in the tower. These tower walls are of reinforced concrete, and of double curvature, giving a flowing shape in vertical section, and circular in cross-section. The fan, if of the usual aeroplane propeller type, with two or more blades of

aluminium or other light material, gives a greater efficiency than a runner with a number of blades. The fan is fitted in or about the narrowest part of the tower, whether of the special type, as above, or any other, and its axis of rotation is co-axial with the tower. With this device, the tower can also be used for freeing gases from water picked up in manufacturing processes. J. F.

860. Distilling Petroleum. E. H. Leslie and E. M. Baker. U.S.P. 1,730,891. Oct. 8, 1929. Appl. Mar. 20, 1923.

The oil to be distilled is raised to a predetermined temperature in a pipe still and delivered on to a spreader in a main vaporiser. Residual liquid, freed from vapour in a lower chamber filled with packing material, is accumulated in the bottom, its exit to the heat exchanger being controlled by ball float valve. The vapours, freed from liquid in an upper packed chamber, are passed directly to the fractionator, situated vertically above the vaporiser, and comprising a number of sections each filled with packing material, whereby the ascending vapours are brought into intimate contact with descending reflux. A level of liquid is maintained in each section by means of overflow pipes which also convey vapours upward. Vapours from the top of the fractionator are totally condensed and the whole of the liquid is returned as reflux. Condensate or vapours may be withdrawn from any section, and condensate may be bypassed to any lower section. Reflux is trapped at the base of the fractionator and may be passed through an auxiliary pipe still to a second vaporiser from which liquid is withdrawn, vapours being returned to the base of the fractionator. Steam and/or petroleum liquid other than the reflux may be introduced to any desired section.

Oil heated to 400–500° F. in the initial stages of the pipe still may be passed to a pre-vaporiser, from which evolved vapours are led to the base of the fractionator, residual liquid being returned to the pipe still for further heating. This arrangement permits an increased rate of heat transfer. L. H. A.

861. Old Stills Give Way to Ultra-Modern. *Oil and Gas J.*, 1929, 28, 18, 76, 77, and 157.

The article describes modern pipe still and bubble tower equipment installed by the Pennsylvania Refining Co. to supersede a battery of crude oil shell stills. The modern plant, which is compact and flexible, comprises a topping unit, a vacuum unit, and a re-run unit.

Topping Unit.—The crude oil preheated to 350° F., is raised to 580° F. in the pipe still, which is comprised of the usual convection and radiant sections. The hot oil is fed on to the fourth bubble cap tray of a tower 84 ft. high and 4 ft. 3 ins. diam., containing 23 bubble cap trays. Above the top tray a short section packed with Raschig rings permits temperature control of the tower by means of liquid pumped on to it from the top tray through a heat exchanger and a regulated cooler. The bottoms are steam-stripped on the lower trays; the ascending vapours are fractionated so that light benzine passes overhead, while three side-streams are taken from suitably selected trays. These cuts are steam stripped in an auxiliary tower comprising three non-communicating sections, each containing five bubble cap trays; each side stream is introduced at the top of its appropriate section and leaves from the bottom, steam being admitted above the bottom plate of the section. The stripped side streams are taken off as special naphtha, kerosene, and gas oil; vapours liberated by the steaming are returned to the main tower at the appropriate points.

Vacuum Unit.—The hot bottoms from the topping unit at about 470°F. are pumped directly to the pipe still of the vacuum unit where the temperature is raised to about 670°F. This oil is then fed to the mid point of the vacuum

tower, the upper section of which is of greater diameter than the lower section. The pressure is reduced to 35-40 mms. mercury by means of a barometric condenser and air ejector; steam superheated to 550°F. is admitted to the bottom of the tower at the rate of one-eighth to one-quarter lb. per barrel of oil charged. Temperature control of the tower is effected by a reflux condenser cooled by preheated crude oil. The overhead is a readily pressable wax distillate; a side stream for dewaxing by dilution and centrifuging is drawn just above the hot oil inlet; the bottoms are run to storage as steam refined stock. These products are withdrawn from the vacuum by centrifugal pump.

Rerun Unit. This is designed to recover the naphtha from centrifuged solution in the manufacture of bright stock, but may be used to run pressed distillate to produce neutral oils. It resembles the topping unit in general design, but has a reflux condenser cooled by the charge and has no auxiliary stripping tower. Superheated steam is admitted to the base of the tower. Two side streams may be taken off in addition to overhead and bottoms.

Control is centralized so that the three units may be run by one man. Along one side of the control room is the "receiving house" consisting of look glasses and sampling cocks, for examination of the quantity and quality of the products, and the manifold valves for the disposal of the latter. Along the opposite side is a control panel containing every indicating and recording instrument for each of the three units, together with all control valves.

L. H. A.

862. Apparatus for Recording Progress of Gas Reactions by Pressure Drop. Anon. *Oil & Gas J.*, 1929, **28** (14), 133.

The apparatus includes the use of balanced electrical oscillating and resonating circuits and utilizes changing condenser capacity to denote pressure changes due to movement of a diaphragm within the reaction chamber which accentuates the movement of one of the two plates of a variable condenser. One side of a gauge is filled with the reacting gas mixture and the other with an inert gas, and the diaphragm adjusted to its undisplaced position by means of a small levelling bulb. The oscillator is started and a standard 1500 $\mu\mu$ variable condenser increased until a sharp increase in plate current is obtained. The condenser is decreased and the operation repeated, the difference observed being directly related to the pressure drop. The apparatus is standardized by suitable means against known pressures.

H. S. G.

863. Recovering Products from Fractionating Condensers. L. W. Snell. *U.S.P.* 1,725,219. Aug. 20, 1929. Appl. Aug. 23, 1926.

The invention provides a method of obtaining fractional liquid products having predetermined initial boiling points and end points from a crude oil. The oil is vaporized in a pipe still and discharged into the lower end of a vertical fractionating column, where the higher boiling fractions will tend to condense in the lower portion, the lower boiling fractions condensing near the top or passing overhead to the final condenser. The fractionating column is divided into a series of zones one above the other, each consisting of a number of horizontal trays provided with a number of vapour pipes and a liquid sealed overflow. The vapour pipes each have small openings below the liquid level and are partitioned off in an arc so that the relatively cool condensate on the plate has to flow around each pipe before reaching the overflow pipe to the plate below. Condensate percolating through the holes into the vapour pipes is caught up and intimately mixed with the vapours. The vapour and overflow pipes are arranged on alternate sides of adjacent trays.

Vapours entering each zone by-pass the bottom few trays which are reserved for steam stripping of the condensate. The condensate is withdrawn from the bottom plate, passed through a cooler, and a certain portion returned to the top plate of the next lower zone by means of a thermostatically operated valve. In a similar manner the requisite amount of condensate is admitted to the top plate of the column from a cooler trapping part of the vapours passing overhead. Thus a predetermined vapour temperature can be maintained at each end of each zone, with the exception of the lower end of the bottom zone, from which the residual product is discharged.

Practically all of the condensate may be withdrawn from each zone in this way without overheating the column at any point, but if desired the reflux from one zone may be allowed to overflow directly to the one immediately beneath it. L. H. A.

864. Distillation of Petroleum and the Like. P. G. Ward and J. Mitchell. *E.P.* 320,135. Oct. 17, 1929. Appl. April 14, 1928.

An apparatus where in the latent heat of steam is used for the separation of the more volatile constituents of the oil and the latent heat of mercury for the separation of the less volatile constituents. Distillation is effected in a battery of evaporators connected in series, the incoming oil being preheated in a series arrangement of heat exchangers by condensates or vapours.
W. S. S. C.

865. Distilling Hydrocarbon Oils. R. E. Wilson, assr. to Standard Oil Co. of Indiana. *U.S.P.* 1,731,479. Oct. 15, 1929. Appl. Jan. 15, 1925.

The basic principle of the invention lies in an attempt to render a larger proportion of the latent and sensible heat of the vapours, evolved, for example, from a battery of shell stills, available for preheating the oil to be distilled.

The apparatus comprises a fractionating column provided with fractionating plates of any suitable type; in the upper portion of the column a plurality of closed flat helical coils is situated, similar ends of these coils being connected to a manifold with the necessary valves and by-passes, so that relatively cool oil may be circulated through the coils in series, in parallel, or in multiple. At a suitable point in the cooling coil section a horizontal tray of diameter slightly less than the tower is mounted so as to receive practically all condensate from the higher coils. A number of nipples permit upward flow of vapour through the ray; a central well in the tray collects condensate which is withdrawn through a seal and led into the overhead vapour line to the condensers.

The column receives the vapours from a shell still, into which is fed the oil preheated in the helical coils. The vapours rise countercurrent to reflux thrown down by coils situated below the draw-off pan, which reflux may be either returned to the still or taken off as a distillate product. L. H. A.

Engineering.

866. The Junker Diesel Engine for Automobile Use. *Automobile Engineer.* 1929, **19**, 341–343.

Two standardized designs of opposed piston two-stroke Junker engines are described. The smaller develops 45 b.h.p. at 1100 r.p.m. in two cylinders each with two pistons, and the larger size develops 70 b.h.p. at 1400 r.p.m. in three cylinders. Scavenging is effected by means of air from auxiliary pistons operated directly from the upper main pistons, and the available volume of scavenge air is about 1.5 times the main cylinder swept volume.

The scavenge ports are situated at the top, and the exhaust ports at the bottom of the main cylinders, the combustion space being comparatively deep and cylindrical. Fuel is injected to each cylinder from a separate pump through an open type fuel valve, the quantity being varied by altering the stroke of the pump plunger through a conventional foot pedal. The amount of air admitted to the scavange pumps is altered to suit the load and speed conditions, control being divided between the pedal operating the pump stroke and an additional control cock. Test bench fuel consumptions are given showing a minimum consumption of .382 lb per b.h.p. hr. at 39 b.h.p. and 1000 r.p.m. on the two-cylinder design with a consumption of .397 lb. per b.h.p. hr. at 29 and 56 b.h.p. R. S.

867. PROGRESS IN KNOCK TESTING. E. Bartholomew. *National Petroleum News.* 1929, **21**, 42, 43.

The author ascribes the failure of various types of test engine to give the same anti-knock rating on a given fuel when compared with blends of standards, to differences other than faulty technique. In such cases each evaluation is correct for the test conditions established, and it becomes necessary to control laboratory tests in such a manner that they reproduce the conditions in the actual engines in which the fuels are to be used, subject to the limitation that all tests must be at the mixture strength which gives maximum knock.

The development of the Delco type of testing set is described. The first plants consisted of slightly altered air-cooled lighting units with an increased compression ratio and with the head modified to take a bouncing pin element. Speed was not precisely controlled, and even air draughts in the laboratory affected results. Carburetter feed from a float chamber at a constant level was used with a change-over device for running on different fuels. The first important modification was to use water cooling, when it was found that the speed required to be reduced to obtain sufficient knocking. The float chamber of the carburetter was replaced with a constant level device with overflow control, and later the cooling was modified to the evaporative system to give a steady discharge temperature and thus to eliminate one control. The speed of operation was then further reduced as this gave steadier values, and following this the feed chambers were again altered so that the mixture strength could be adjusted to that giving maximum knock. In the early part of 1928 it was found that on changing over from one blend to another of a different nature the knocking intensity was liable to increase suddenly and only slowly to become stable. This necessitated a modification of routine to insure stability before readings were taken.

Comparative tests of ethyl doped aviation gasoline in two different engines led to the discovery that with this anti-knock, different engines gave different ratings, and that, compared with benzole blends, the lead dope was more efficient at high jacket temperatures.

A new design of engine was then developed in which it is possible to test at temperatures approximating to those of air-cooled aero engines. The maximum bore suitable to avoid pre-ignition was found to be 2¼ in. The cylinder and head are evaporatively cooled with a suitable medium such as ethylene glycol. The hand speed control has been eliminated by the use of a belt-driven synchronous generator which floats on the power line of the laboratory.

Tests between 345° and 402° F. jacket temperature, using blends of Mid-Continent gasoline with benzole and ethyl fluid with aviation gasoline did not match at both temperatures. At the lower temperature 1·35 c.c. lead tetraethyl was required in aviation to match a 40 per cent. benzole blend in

Mid-Continent gasoline. At the higher temperature it was found that only ·55 c.c. was required. In some high-temperature tests it was found that benzole had very little better anti-knock value than ordinary Mid-Continent gasoline. Research is now in progress to determine the correct temperature conditions in the test engine to represent the various classes of practical requirements. These tests are being made on cars with special equipment and, owing to the difficulty of precise control and the fact that only audibility tests are practicable, the accuracy is estimated at about ·25 c.c. lead tetraethyl. The author summarises the requirements of a spirit-testing engine as follows:—

1. To be suitable for the reproduction of any practical conditions.

2. Volumetric efficiency to be high to keep the compression ratio as low as possible.

3. Overhead type valves preferred on account of stability and ease of fitting the bouncing-pin indicator.

4. Uniform cooling essential.

5. Piston should be small and preferably of aluminium alloy to reduce the temperature.

6. Cooling should be evaporative.

7. The carburetter should be of the single jet type with means for mixture strength control.

8. The design should be such as to reduce the unsteady effect after change over of fuel as much as possible.

9. The speed should be kept constant at not more than 1,000 r.p.m., and preferably at not more than 600 r.p.m.

10. The displacement should be small to permit of the use of small samples.

11. The working parts should be stiff enough to withstand long periods of heavy detonation.

12. Mechanical means should be provided for measuring the intensity of detonation and also combustion chamber temperatures.

13. The machine should be simple and cheap. R. S.

868. Horsepower Correction for Atmospheric Humidity. D. B. Brooks. *S.A.E. Journal.* **25**, 3, 277–283.

Tests were made on two engines, the first being a six-cylinder three-port overhead valve type, 3½ in. bore and 5½ in. stroke with jacketted manifold, and the second a lorry engine 4¼ in. bore and 6 in. stroke having four cylinders and L head with two side valves. The manifold on the second engine was not jacketted, but was arranged with a hot spot. This engine was used at three compression ratios, fuel being selected to suit the ratio used except in one test during which heavy detonation was allowed at maximum spark advance.

Humidification was obtained by passing steam and cold air into a mixing chamber and thence over a heater. Humidity was measured on the first engine by passing a part of the air over a pair of wet and dry bulb thermometers graduated to ·2° F. On the second engine the thermometers were placed in the main supply line, thus introducing a small error due to evaporation from the wet bulb. All measurements of humidity are expressed as pressure of water vapour in millimetres of mercury. The tests were made over a range of air fuel ratios, and all results are in close agreement with two exceptions. In one case power increased with humidity owing to the use of

a weak mixture. The effect of added water in this case was to increase the effective mixture strength. In the other case the loss of power with humidity was smaller than usual, this being the test with heavy detonation in which the anti-knock effect of the water vapour partly offset the loss measured with non-detonating fuel. It is concluded that failure to correct for humidity may lead to errors of the order of 10 per cent. of i.m.e.p. under extreme conditions; humidity under all normal conditions may be corrected for by deducting the observed pressure of water vapour from the barometric pressure. In correcting engine performance data at different air-fuel ratios the fuel flows must be multiplied by the same co-efficient as the power values. Optimum spark advance increases linearly with increasing humidity. The results obtained closely verify the hypothesis that the power output of an engine depends on the oxygen content of the induced charge, water vapour reducing this content. There appear, however, to be indirect effects. Thus, the optimum spark advance increases with humidity, but the loss of power to be expected from this cause is not observed. It is suggested that differences in heat loss, dissociation, and change of specific heats may vary operating conditions, and so compensate for part of the total loss of power anticipated.

R. S.

869. THE USE OF STEEL IN THE OIL INDUSTRY. R. Hadfield and S. A. Main. *Journ. Inst. Petr. Techn.*, 1929, **15**, 611-633.

With regard to well-boring tools, the importance of using the best steel is stressed, and it is shown by microstructure that the lowest practical forging temperature is desirable. Forging must be completed at above the critical temperature (circa 700° C.). Heat treatment after forging removes strains and effects a fuller refinement of the structure than can be obtained under the best forging conditions. In hardening by quenching, heating, preferably by lead or salt bath, should be even and the quenching medium should be vigorously circulated. The optimum quenching temperature is just above the critical temperature and varies with different types of steel. The average footage of bits hardened under temperature control was 13 ft. 1½ in. and the rate of drilling 13 ft. per min. compared with 8 ft. 6½ in. and 9 ft. 11½ in. without such control. Alloy steels (Vd. 0·20per cent. and Cr. 0·60 per cent.) improve drilling bits and cutters, particularly from the point of view of toughness. The superior qualities of intermediate manganese steel (C. 0·35–0·40 per cent., Mn. 1·5–1·75 per cent.) are exemplified in a table of results where it is shown that plain carbon steel is inferior with regard to yield point and toughness. Bits with hard cutting material (Tungsten, Molybdenum Carbides, alloys of Iron with Tungsten, Chromium and Manganese, and Cobalt-Chromium alloys) are able to withstand drilling shocks without shattering. The introduction of the weldless jar has enabled alloy steel to be used for its construction, while manganese steel has proved to give increased wearing when used for drive chains and sprockets.

In oil refining processes necessitating the use of high temperatures in conjunction with high pressures, the use of heat-resisting steels is imperative. A specimen of this steel had at 800° C. a tensile strength nearly four times that of ordinary steel, while its heat scaling was limited to the formation of a slight surface film. Where non-scaling properties have been developed to the highest degree, strength and toughness at high temperatures are liable to decrease. Two heat-resisting steels (high percentage nickel-chromium type) are described which are suitable for the manufacture of cold-drawn tubes, recuperator castings, Bergius coal-hydrogenating reaction vessels, etc., and are resistant to sulphurous gases. With regard to the latter, a heat-resisting steel has been developed which contains no nickel

and is ideal for furnaces for the roasting of zinc ore and mechanical stokers. The absence of nickel renders the castings somewhat brittle, but the material is forgeable and the production of hot rolled tubes is practicable. Due to increased working temperatures (400°–600° C.) in steam practice, ordinary steel shows a propensity to creep under load and rapidly becomes weaker with increasing temperature. Here, heat-scaling troubles are not serious and a steel of the nickel-chromium-molybdenum type has been developed with a creep stress limit of 17 tons/sq. in. at 450° C. and 6 tons at 550° C. It does not become brittle on repeated heating and cooling and has a coefficient of expansion substantially the same as that of ordinary steel. In conclusion, the authors emphasise the importance of close collaboration between the user and steel manufacturer in all questions of special importance where steel is employed. W. H. T.

870. Oil Runways for Air Ports. J. A. Herliky. Eighth Annua Asphalt Paving Conference, October and November, 1929.

The purpose of the paper is to emphasise the necessity for paved runways for air ports and to describe their construction with reference to asphalt pavings. The main reasons for the necessity of paved areas are, firstly, to facilitate the operation of heavy planes during wet weather, and secondly, to eliminate the dust nuisance. As it is impracticable to pave the whole air port area, paved strips must be constructed as taxiways and runways. For landing, these should be 120 ft. wide and for taking off 90 ft. is sufficient for width. The length varies with the altitude above sea level; at sea level 2,500 ft. of clear field including 1,500 ft. of paving, and at 5,000 ft. above sea level, 4,000 ft. of runway including 2,500 ft. of paving. The chief disadvantage of oiled runways is their difficulty of illumination at night. Hard runways are desirable as take-off strips, but for landing a more resilient surface is required. The methods of producing the former are the same as for highway work, but the preparation of the latter presents a different problem. Here, the first process is to examine the soil and adjust it by suitable admixture to give a fairly uniform grading from 200 mesh to $\frac{1}{2}$ in. The thickness of runway surface will depend largely on local conditions. After a thorough harrowing of the soil, the road oil is applied through a force feed spreader in separate applications of $\frac{1}{4}$ gal. per sq. yd. A suitable oil would be a 60–70 per cent. asphalt oil of 80 penetration at 77° F. After each application, the runway is again harrowed to ensure thorough mixing of oil and soil. Normally, the total application is $\frac{1}{2}$ gal. per sq. yd. per inch of thickness. After all the oil has been applied, it is mixed by means of a blade grader with the blades set at 45 deg. with the direction of travel. It is necessary to blade the mixture from 20–30 times to get a thorough combination of the mixture which should have a final uniform dark chocolate appearance and tend to "crawl" when disturbed with a stick. Final levelling is carried out with a straight blade followed by rolling with a light roller or preferably by a truck with large pneumatic tyres. This method of construction gives a smooth, tough and resilient surface which can be built for less than half the cost of any other form of paving. W. H. T..

871. Operation of Diesels in West Texas. Humble Pipe Line Co. *Oil and Gas J.*, 1929, **28**, (15), T. 90.

The two major causes of trouble in connection with Diesel engine operation in West Texas are fuel and water. The fuel used is in nearly all cases crude as it comes from the field. It often contains as much as 8 to 10 per cent. by volume of hydrogen sulphide, together with some water and solid matter. Early engines suffered from fuel valve and pump plunger troubles which

led to the use of special materials, and sometimes to partial redesign of fuel systems. Later excessive liner wear developed, with fouling of exhaust valves, deterioration of fuel lines, etc. A comparison between engines using sweet and West Texas crude showed the latter to cause more than four times the cylinder wear in a given time. A 14½ in. by 17 in. cylinder running at 200 to 275 r.p.m. only ran 232 hrs. per 0·001 in. wear. Later again serious crankshaft corrosion was discovered on an engine running on West Texas crude. The installation of a continuous reclamation system for the lubricating oil overcame this difficulty. Cooling water must be analysed periodically for hardness and treated if necessary. The zeolite process is good, but is not satisfactory when there is iron in the water. Closed jacket systems may be needed in extreme cases. Sand storms are a source of trouble, and good air filters are essential. A case is reported in which half a shovel of sand was removed from an intake pipe during an overhaul.

R. S.

872. Diesel Engines in Main Line Stations. R. G. Lewis. *Oil and Gas J.*, 1929, **28**, (15), T.68-74.

The Diesel engine is preferred for pipe-line systems both on account of fuel economy, which is in the ratio of 1 to 3½ when compared with steam plant, and because of the ready availability of the fuel. Waste heat may be used for heating the engine room and also for heating lubricating and fuel oils. The life of a modern engine is at least 30 years, and the upkeep costs fall within 3 per cent. of the cost of a new engine. The various types of heavy oil engine are described, preference being given to the closed type of fuel valve for air injection engines, and to cold start solid injection engines. Gravity methods of lubrication are only satisfactory if carefully installed and maintained. Oil filtration by means of strainers has been superseded either by chemical treatment, or preferably by centrifuging. Water cooling requires the use of soft water to avoid cracking of cylinder heads. If reservoir cooling is adopted the capacity should be about 420 gals. per h.-p. Closed cooling systems are frequently adopted. In these a limited supply of water circulates through the engines, this water being cooled by passing it through jacketted cooling coils. Small engines are sometimes cooled by means of radiators and fans. Fuel may need to be centrifuged if it is dirty at source. Without this precaution there is risk of liner wear and fouling of fuel and exhaust valves. The general tendency is towards the use of larger cylinders with larger crankshafts and bearings. Operators must be properly trained to get the best results. Air filters are being more generally used with beneficial results as regards cylinder wear. Pyrometers in the exhaust outlet branches permit of rapid diagnosis of trouble in any cylinder. Chromium plating has resulted in elimination of corrosion of fuel pump and valve parts. Crankshaft alignment must be checked at regular intervals, and any necessary adjustments made to bring dropped bearings back to position.

R. S.

873. Past and Future of Electric Welding. W. M. Murphy. *Oil and Gas J.*, 1929, **28**, (15), T.118-121.

Early attempts to construct arc welded pipe-line joints were characterised by high cost owing to rapid changes in design which prevented quantity production of the apparatus. Modern methods show lower costs per weld than other methods of jointing provided the plant can be kept in continuous use. Unsuitable and non-uniform welding rod has been replaced by carefully-selected and tested material containing suitable flux to absorb impurities, and the tensile strength of the finished weld can be guaranteed to be 80 per cent. of the pipe stock—the majority of joints being stronger

than the plain pipe itself. Brittleness and porosity, regarding which there were frequent complaints on early lines, have been eliminated by the use of proper welding rods and the correct training of the operators themselves. Tie-in welds have been made satisfactory by change of welding heat and the use of one or more additional layers of deposited metal. Joint design has been considerably improved in order to give freedom from leakage, stronger joints, greater flexibility, and greater ease for repairs. With suitable organisation of labour, under fair conditions, an experienced welder can make at least 35 welds per nine-hour day on 8-in. pipe, and 18 welds on 20-in. pipe. R. S.

874. Automatic Electric Switch made at Low Cost. *Oil and Gas J.*, 1929, **28**, (16), 131.

Electric contact is made by tilting a partly filled bottle of mercury, thus allowing the mercury to roll down and fill the space between two contact points to complete the circuit. The bottle is tilted by the rotary movement of a steel spring as the metal is heated or cooled. The fineness of control is dependent upon the length of the spring, the coefficient of expansion of the metal and the precision used in assembling the moving parts.

The frame is a piece of iron pipe one end of which is threaded and fastened into an ordinary outlet box. The other end is cut to leave a short projection, and to this one end of the spring is bolted or welded. The box and pipe are held together by two thin lock nuts. A steel or brass rod, $\frac{1}{4}$ to $\frac{1}{2}$ in. diameter, runs through the centre of the pipe. One end is fastened to the loose end of the spring, and the other is threaded to take a small nut that is put on leaving about 1 in. of the threaded rod exposed. A disc is threaded on to the rod and welded to the nut and rod. The face of the instrument is next slipped on to the rod and a nut holds it against the disc to form a friction contact whose tension may be varied as required.

A small bottle is mounted on the face, a few drops of mercury are sealed in the bottle and as the bottle is tilted, by movements of the face, this mercury rolls from one end to the other, making or breaking contact between two copper terminals which are sealed into one end of the bottle and are placed about $\frac{1}{4}$ in. apart.

The spring must be heavy enough to exert a positive force on the rod and rigid enough to withstand adjustment of the face. Five turns, $\frac{1}{4}$ in. in diameter, of ordinary steel spring will operate the instrument for a difference of 10° of control. For finer control a longer spring is necessary. W. L.

875. A Chemical Carburettor. The "Catalex." *Rev. Pétrolifère*, No. 340 (Oct. 5, 1929), 1417.

The essential feature of the "Catalex" carburettor is the use of catalytic metal surfaces, heated by exhaust gas, over which the mixture of fuel vapour and primary air is drawn. The partially oxidised mixture is then stabilised by the admixture of cold secondary air and the resulting charge ignited in the cylinder in the usual manner.

It is claimed that this procedure causes a partial cracking of the fuel, giving a product with high anti-knock value; also that the power output of an engine is increased by the more rapid combustion of the partially oxidised charge. There is little or no heat loss from the fuel in the carburettor since the mixture is preheated there and the initial reactions are endothermic. This carburettor enables the use of gas oil as a fuel for ordinary petrol engines provided spirit be used for starting. The gas oil gives products which burn without coking the plugs or combustion space. The saving in running costs is considerable. G. B. M.

876. METALS FOR HIGH TEMPERATURE SERVICE. C. M. Ridgway. *Oil and Gas J.*, 1929, **28**, (20), 192, 305, 308, 312.

The operation of cracking units at temperatures over 1000° F., and pressures over 1200 lb./sq. in. call for care in the selection of materials. The engineer is chiefly concerned with (1) primary physical properties, *e.g.*, elastic limit; (2) secondary physical properties, *e.g.*, coefficient of expansion; (3) chemical properties, *e.g.*, corrosion resistance. The primary physical properties of metals are usually defined as ultimate tensile strength, yield point, elastic limit, proportional limit, creep limit, reduction of area, elongation, impact, and fatigue stress.

The proportional elastic limit and the yield point decrease with increasing temperature, but the ultimate tensile strength increases to about 600° F. The ductility, as measured by elongation and reduction of area, decreases to about 600° F. and then gradually increases, while the resistance to shock and impact increases to about 400° F., decreases to 1000° F., and again increases to about 1500° F. Physical properties at room temperatures are no criterion of those at elevated temperatures. Under static loads, below creep limit, elongation is elastic at first and then plastic until the resistance to slip balances the load. Under greater loads, fraction occurs. Under 900° F. the proportional elastic limit is lower than the creep test, and therefore the elastic limit should be used for temperatures below this figure.

For pipe-still tubes a carbon content of 0.2 to 0.3 is desirable in plain carbon steels.

Carbon steels show minimum brittleness at 210–400° F., and maximum at 750–930° F., their strength decreasing rapidly after 600° F. is reached.

Short time tests at elevated temperatures do not represent actual behaviour under continual stress. Carbon steels with less than 0.15 per cent. of carbon give trouble due to grain growth at high temperatures. The physical properties of steels at high temperatures can be improved by the use of certain alloys, such as manganese, nickel, chromium, molybdenum and tungsten. G. S.

877. METHOD FOR REGULATING MIXTURE TEMPERATURE, MIXTURE STRENGTH, AND COOLING OF INTERNAL COMBUSTION ENGINES. J. Szal and P. Z. Lotnicze. E.P. 316,783. Aug. 13, 1928.

A connection is made between one of the working cylinders of the engine and a controlling valve driven from the engine. The pressure in the working cylinder varies with altitude or throttle opening and is transmitted through the controlling valve to a piston in a relay cylinder. This cylinder is oil filled and the oil transmits pressure along suitable pipe lines to smaller cylinders and thence through their pistons to linkage controlling mixture strength, mixture temperature, and cooling. The mixture-strength control acts on the carburettor, the cooling control on adjustable shutters in front of the engine air cooling fins, or in front of the radiator if the engine is water cooled, and the mixture temperature control on an exhaust heater by-pass valve.

An alternative method makes use of a separate cylinder instead of a working cylinder from which to take the main control connection. R. S.

878. EFFECT OF A CENTRIFUGAL SUPERCHARGER ON FUEL VAPORISATION. C. F. Taylor. *S.A.E. Journ.*, 1929, **25**, 49.

The effect of a high-speed centrifugal supercharger on fuel atomisation and distribution was investigated with a Chrysler 1926 six-cylinder engine connected to an electric dynamometer. In addition to the usual carburettor, a change-over carburettor fitted with a high-speed blower was employed. To avoid obscuring comparative results the induction manifold jacket,

normally heated by the exhaust, was replaced by an air heater at the intake end; this enabled the heating due to the blower to be simulated in the unblown charge. The two carburettors were identical in design and the length and shape of the passage between the carburettors and the induction manifold were made as nearly the same as possible except for the presence of the blower.

Comparative runs were made at given speeds and loads, engine conditions, particularly the temperature and pressure in the induction manifold, being held the same in the two cases. Under normal operation the supercharger would give an increased pressure and temperature in the manifold, but these were compensated for by throttling the blown charge and heating the air to the unblown carburettor respectively. Runs were made on U.S. commercial petrol at 1000, 1500, 2000 and 2500 r.p.m. at full, three-quarter and half-load. The word "load" in this paper refers to the lowering of pressure in the induction manifold. Thus, "full load" corresponded to the depression in the manifold at full throttle with the unblown arrangement. The method was, at each "load," to vary the fuel flow and read the dynamometer load required to maintain a constant speed. From these results curves were plotted showing the variation of B.M.E.P. and specific fuel consumption with engine speed, with and without the supercharger.

It was found that, at full "load," there was a slight increase in power and a slight reduction in fuel consumption due to the better mixing and atomisation with the blower. At three-quarters and half load the difference was negligible with this engine. A less volatile fuel, however, containing 50 per cent. kerosene, showed a more marked improvement with the blower.

A second series of tests, in which only the carburettor with supercharger was used, showed that an increase in blower speed from 7000 to 21,000 r.p.m. steadily increased the performance and decreased the fuel consumption. The blower speed throughout the first series was maintained at 16,400 r.p.m. The temperature of the charge on the induction manifold was maintained constant by means of an air heater at the inlet end so that the effect of blower speed on atomisation and distribution only should be observed. The blower was electrically driven for both series of tests but the power absorbed was apparently very small. It would probably be more than balanced, on normal operation, by the better vaporisation due to the heating of the charge in the blower. G. B. M.

879. Balance of Furnace and Fuel Costs Determines Optimum Stack Temperatures. S. D. Turner. Reprinted from *Chem. and Met. Eng. Refiner*, 1929, **8** (9), 82, 86.

Steam boiler efficiencies, justifying low stack temperatures, are not ideal for furnaces employed in petroleum processes, although often they are assumed to be. In the latter any saving derived from the use of low stack temperatures is counterbalanced by the cost of the larger heating surfaces required to produce this low temperature.

For a given heat output there is an economic balance between the cost of the fuel and the cost of operating and maintaining the heating surface. The formula derived from first principles and illustrated by a worked example shows that the rate of heat transfer from the hot gases to the tubes is equal to the ratio of the cost of the charge and the cost of pumping it over the heating surface to the cost of the fuel.

From the rate of heat transfer the stack temperature can be calculated. For these furnaces the stack temperatures so calculated are considerably higher than those for steam boilers calculated along similar lines with steam boiler data. This does not necessitate large furnaces merely to obtain a low cost for unit-heating surface.

The capacity of the furnace being determined by the area normal to the path of the hot gases, the maximum rate of firing withstandable by the lower tubes is calculated, and sufficient tubes installed above these to cool the gases to the theoretical value calculated. J. M. P.

880. Combustion Chamber Design in Theory and Practice. W. A. Watmough. *Automobile Engineer*, 1929. **19**, 329-339.

The author illustrates and describes a large number of cylinder head designs, including "T" and "L" heads, the latter with various degrees of turbulence and controlled flow, and gives curves to show that improvement in brake mean pressure follows reduction of pumping losses and also that control of flow and avoidance of over-turbulence smooths the rate of pressure rise during combustion. The design of the combustion chamber for best results is stated to be inseparable from the design of the engine as a working unit, heat control in both the head and the induction system being required to give the flexibility, smoothness, power and economy required in service. R. S.

881. Anti-Knock Compounds. W. Botte. *Rev. Pétrolifère*, Nos. 336-343 (Sept. 14-Oct. 26), 1929.

In this series of papers the author reviews the theories which have been advanced to account for "pinking" in the internal-combustion engine and the experimental work on which they are based. He shows how the inadequacy of chemical analysis as a means of rating spirits with regard to their pinking tendency has led to the development of engine tests. A description is given of a number of engines and experimental methods now in use in different countries. The necessity for standard methods and standard reference fuels is stressed and various possibilities are discussed. A list of anti-knock compounds is given together with the preparation and properties of the more widely used compounds. G. B. M.

Anti-Detonation.

882. The Movements of Flame in Carbonic Oxide-Oxygen Explosions. H. B. Dixon. *Nature*, 1929, **124**, 580.

This memoir describes some recent researches by Bone and Fraser (*Phil. Trans.*, 1929, 228A) on flame-photography of carbon monoxide Knall-gas ($2CO_2+O_2$).

In the first place the effect of varying degrees of mixture dryness was studied. The progressive removal of moisture by contact with P_2O_5 was found—

(*a*) to slow down the initial flame movement;

(*b*) to increase the luminosity of the initial flame;

(*c*) to prolong the afterburning;

(*d*) to make the combustion less complete.

These mixtures were fired in a tube of moderate length. In a longer tube the initial flame in very well dried mixtures wavered and died out before reaching the end of the tube. This flame extinction could be avoided by the action of an electrostatic field in the path of the combustion. It was also noted that, in all mixtures, combustion was completed initially in the gas around the spark-gap, as this region remained dark when the afterglow was filling the rest of the tube.

The effect of very powerful condenser discharge sparks as the source of ignition was to give more instantaneous ignition and faster initial flame speed than with less intense ignition sources.

By an ingenious device the authors were able to study the effect of shock-waves which overtook and passed through the initial flame. Such shock-waves gave rise occasionally to "auto-ignitions" in the unburnt gas ahead of the flame. A photograph, showing this effect in a mixture about to detonate, shows also the banded structure now known to be due to the helical motion of the flame. G. B. M.

883. Explosive Limits of Industrial Gases. J. Yeaw. *Ind. & Eng. Chem.*, 1929, 21, 1030.

The object of the experimental work was to obtain the widest explosive limits, or the least amount of air or oxygen (upper limit) or the least amount of gas (lower limit) which could make a gas-air mixture dangerous. The relation between the composition and the explosive limits of widely varying complex inflammable gas mixtures, generally encountered in the manufacture of city gas is shown. These gases can be classed, on the basis of explosive limits, into three groups: (1) those which contain illuminants and have a high percentage of combustible; (2) those which do not contain illuminants but have a high percentage of combustible; (3) those which do not contain illuminants and have a low percentage of combustibles. Even a small percentage of combustibles reduces the upper limit from about 70 per cent. to about 30 per cent. but the lower limit is only slightly affected by as much as 7 to 8 per cent. of combustibles. Great changes in the ratio of hydrogen to carbon monoxide do not affect the upper limit but increases in the percentage of methane tend to lower the upper limit of the mixture.

When the percentage of inerts is raised, the amount of combustible at the lower limit is reduced and this lower limit rises, but at the upper limit the inerts tend to dilute the mixture and promote more complete combustion, thus keeping the upper limit high.

The explosive limits of all the gases have been calculated from their analyses by means of a formula first presented by Le Chatelier and later amplified and developed by Coward, Jones and others. If a, b, c are the relative proportions of its components and A, B, C are their respective explosive limits then L, the limit for the mixed gases, is given by t' e equation

$$L = \frac{100}{\frac{a}{A} + \frac{b}{B} + \frac{c}{C}}$$

These calculated values agree with those found experimentally remarkably wel and not only show that this work checks that of others in the field, but also gives further proof of the value of such a means of determining dangerous conditions. W. L.

Cracking.

884. The Carburol Cracking Process. K. Bender. *Petr. Zeitschr.* 1929, 25, 1187-1191.

The deposition of coke on the walls of the heating tube and of the cracking vessel in the usual cracking processes renders them discontinuous. The present carburol cracking process is continuous and no coke formation occurs. The oil to be cracked is passed under pressure to a preheater thence to cracking coils through which the oil passes with a definite velocity. It is brought to

the required temperature and kept at this temperature for some time during which benzine forms. The pressure of the oil is then released by means of an expansion valve and cooled by introducing fresh oil. The high-boiling constituents are cooled further, and the heat obtained therefrom is utilised in cracking fresh oil. The viscous residues pass to a residue cooler, whilst the light vapours are conveyed to a Raschig tower and dephlegmator, and the benzine vapours collect after cooling in a special tank, and the gas passes to a gasometer. The essential features of this process are the absence of free carbon in the cracking coils, expansion valve and heavy oil separator, after long periods of cracking, the possibility of obtaining the residue in the liquid phase with any desired viscosity. The residue can be used as a crude oil in the manufacture of asphalts or as a normal heating oil according to its viscosity. The cracking gas can be used as Blau gas or in the manufacture of synthetic caoutchouc.

The apparatus is small, and 100 tons of crude material a day can be treated in an oven space of 600 sq. ms. W. S. C.

885. Catalytic Cracking of Petroleum Oils, Tars and the Like. Compagnie Internationale pour la Fabrication des Essences et Pétroles. E.P. 320,286. Oct. 10, 1929.

This is an addition to E.P. 319,341, and comprises the interposition between the catalyser and dephlegmator of a purifier containing divided oxides or metals on a porcelainic or porous carrier, this purifier being maintained at a temperature sufficient to avoid any condensation. W. L.

886. Manufacture of Light Hydrocarbons By Catalytic Cracking of Petroleum Oils, Tars and the Like. Compagnie Internationale pour la Fabrication des Essences et Pétroles. E.P. 320,287. Oct. 10, 1929.

This is a further elaboration of E.P. 319,341 and E.P. 320,286, and comprises the connection to the dephegmators of groups of purifiers for the refining of the light products issuing from the dephlegmators.

These groups consist of a heater, for the light products, followed by one or more purifying chambers, for retaining sulphur compounds, and one or more catalytic reaction chambers and finally condensing apparatus. The non-condensed products are collected in absorbers before the evacuation of the permanent gases. The vaporiser of each main group receives oil heated in successive heaters at a progressively increasing temperature, additional water being supplied as steam. W. L.

887. Catalytic Cracking of Petroleum Oils, Tar and the Like. Compagnie Internationale pour la Fabrication des Essences et Pétroles. E.P. 319,341. Oct. 10, 1929. Appl. Dec. 19, 1928. Conv. (Fr.) Sept. 21, 1928.

This invention comprises a process and apparatus for cracking at atmospheric pressures. Oils are vaporised as completely as possible, and the vapours led to a catalyser and thence to a dephlegmator. Here light products are removed and the residual liquids led to a second vaporiser—catalyser—dephlegmator group, followed in series by other like groups. These groups are arranged to be used in parallel or in closed circuit of any number of apparatuses for utilisation with cyclic permutation. W. L.

888. Method of Cracking Oils. R. H. Crozier. E.P. 319,224. Sept. 12, 1929. Appl. May 18, 1928.

In a vertical retort, in which coal or shale moves downwards continuously, gas mains are located at different levels through which the vapours generated

at different temperatures may be withdrawn. These gas mains terminate in boxes for the collection of liquid fractions, and the vapours are caused to bubble through the liquid before passing to the condenser. The boxes are lagged to maintain them at a definite temperature, and each box is connected to the next lower box with a seal to keep the level constant. The heaviest fractions are thus concentrated, and pass downward to the lowest box, whence they are delivered into the hot coke and again subjected to the cracking action of the retort. The permanent gases produced are returned into the flues and burnt to the greatest advantage with hot air heated by contact with the moving mass of residual coke.

In this manner a large portion of the valueless products are converted into more valuable oils of low boiling point, coke and fixed gas. The residual coke will possess a higher heat value than coke produced by ordinary distillation. R. P.

889. Treatment (Cracking) of Hydrocarbons. C. P. Dubbs. U.S.P. 1,729,307-8. Sept. 24, 1929. Appl. (1) Feb. 26, 1920. (2) Sept. 24, 1923.

(1) Oil is heated to above 800°F. at a pressure high enough to prevent destructive distillation. It is then flashed, the flash chamber being at atmospheric pressure, a substantially complete separation into vapour and coke being obtained. Vapours are fractionated, and all insufficiently converted material is fed continuously to the charge stream.

(2) The residual oil from the reaction chamber of a cracking process is removed and introduced into a coking still. The pressure on the still is released and the oil coked by superheated steam. W. S. C.

890. Apparatus for Pressure Distilling Oils. G. Egloff and H. P. Benner. U.S.P. 1,729,035. Sept. 24, 1929. Appl. Feb. 21, 1921.

This invention relates to improvements to that type of apparatus in which the oil is heated in a series of tubes then passed to an expansion chamber wherein vaporisation takes place and the vapours removed and condensed. The novelty of the present invention lies in the manner of constructing the expansion chamber in order to facilitate cleaning. This is accomplished by making the expansion chamber of two concentric shells, the inner one of which is removable and preferably formed of separate segments. The outer shell has a removable head at one end and the inner shell is connected to this head. The inner shell is mounted on roller bearings so that it can be easily pulled out of the expansion chamber. The segments may then be separated in order that they may be easily and quickly cleaned of the precipitated carbon. R. P.

891. Process of Treating Hydrocarbons. G. Egloff. U.S.P. 1,733,654, Oct. 29, 1929. Appl. Jan. 12, 1920.

A coil is seated in a furnace and a horizontal cylindrical vapour chamber is situated above the coil. This chamber slopes towards the rear of the furnace. The oil passes through the coil and enters the chamber at its forward end, whilst the vapours pass from the rear end through a goose neck provided with a valve into the dephlegmator, which also has a cock at the outlet leading to the condenser. At the centre of the rear end of the vapour chamber an outlet pipe with valve leads to the raw oil pump suction.

Thus gas oil may be subjected to a temperature of 750° F. in the cracking zone, and a still pressure of 100lb. Sufficient fresh oil is supplied to insure that the oil passes through the line from the chamber back to the pump. When a differential pressure is used the still may be maintained at 100lbs.,

the dephlegmator, say, at 75 lbs., and the condenser at 50 lbs. by regulating the valves between sections of the plant. The reflux oil from the dephlegmator returns to the vapour chamber through the goose neck, thence is pumped back through the coil along with raw feed. R. P.

892. Apparatus for Treating Oil. G. Egloff and H. P. Benner. U.S.P. 1,733,655, Oct. 29, 1929. Appl. Sept. 1, 1920.

The object of the invention is to provide an apparatus for simultaneously dehydrating and cracking oils. In a firebox a lower shell and an upper shell are disposed horizontally forming a double still.

The stills are provided at each end with circumferential gear and connected with intermediate gear so that the stills will revolve in the same direction. Each shell is connected at one end with a stationary cap by means of a ground joint or other suitable means. The oil is charged through the stationary part of the lower still and the vapours pass through a pipe projecting along the bottom of the upper still, the connection being made at the stationary parts. The vapours from the upper still pass out into a condenser coil and thence to a receiver. The oil in the lower member is heated by means of a pair of burners arranged at opposite sides of the upper part of the shell and extend longitudinally along the latter. These burners are arranged at or above liquid level in the still to primarily heat the upper surface of the oil charge, thus preventing foaming of the emulsified oil.

Similar burners are used to heat the upper member, the arrangement being made to superheat the vapours. By heating the stills independently the temperature can be regulated to dehydrate the oil and gradually fractionate the vapours without undue heating and separately heat the vapours to cracking temperature. R. P.

893. Process for the Conversion of Hydrocarbon Oil. G. Egloff and J. C. Morrell. U.S.P. 1,733,656, Oct. 29, 1929. Appl. July 24, 1922.

The invention relates particularly to the conversion of petroleum into other compounds such as organic acids, ketones, aldehydes, alcohols and other oxidation products, besides gasoline. The oil is treated in the presence of a catalyst and a regulated quantity of oxidising gas, such as air, oxygen, or oxides of nitrogen, preferably continuously and under a pressure of the generated vapours.

A still is mounted in a furnace, heated by means of gas burners, and connected to a dephlegmator and condenser. The oil to be treated is introduced near the top of the tower and flows down through the rising vapours. The preheated reflux is drawn from the bottom of the tower and discharged, through a perforated pipe, into the bottom of the still, whilst an oxidising gas is introduced at the same time into the oil before entering the still.

The oil in the still is heated to cracking temperature, and the vapours pass out at the top to the dephlegmating tower. Within the still, horizontal pans are spaced upon which is placed the catalyst, for example, copper oxide or barium peroxide. Some of the pans are immersed in the oil and some are in the vapour space so that the action can take place in both phases. Oils such as wax distillate, gas oil or kerosene are suitable for a process of this character. R. P.

894. Art of Cracking Hydrocarbons. E. C. Herthel. U.S.P. 1,733,799, Oct. 29, 1929. Appl., June 11, 1927.

In the cracking of the heavier petroleum oils for the production of gasoline, asphaltic compounds are formed. When a still is charged with gas oil and heated to a cracking temperature, cracking takes place in a gradual and progressive manner, and as the pressure distillate is removed the heavier

hydrocarbons formed mix with the charge. For the most part these constituents remain in solution at first, but the amount of asphaltic constituents increases until saturation point is exceeded. With the exception of certain compounds, the formation of coke on the surfaces of the plant is avoided as long as the asphaltic constituents are kept in solution. There is a relatively small quantity of objectionable substances, apparently sulphur compounds, which, on cracking, preferably deposit on the heated surfaces. In carrying out the invention both the nature and the amount of asphaltic constituents formed in the pressure still are regulated and controlled. Some of these constituents are selectively removed by circulating the oil within the still through a body of fuller's earth or similar absorbent material. The amount of asphaltic substances remaining in the still charge is regulated by discharging pitch-laden oil and supplying raw oil.

The plant consists of a pressure still and a battery of heating tubes. The oil is circulated from the bottom of the still through the heating tubes and discharged into the upper part of the still. The still contains a screen situated below the oil level to support a bed of fuller's earth, and the circulating oil must pass through this to maintain circulation. In a still with an operating charge of about 10,000 gallons the charge of fuller's earth may amount to 4 or 5 tons, 15 to 30 mesh. R. P.

895. Manufacture of Low-Boiling Hydrocarbons. I. G. Farbenindustrie Akt. E.P. 320,473, Oct. 17, 1929. Appl., Aug. 31, 1928, and Nov. 22, 1928.

As an example of the process a topped American crude oil is introduced in a hot state into a high-pressure apparatus and treated with hydrogen in the presence of a tungsten catalyst at 400° C., and 200 atmospheres pressure. About 20 per cent. of the oil is converted into gasoline and light middle oil. The residue is transferred to a second reaction vessel and treated with hydrogen at 450° C. and 200 atmospheres pressure in the presence of a catalyst containing molybdenum and chromium. A further 50 per cent. of the original oil is converted into gasoline and middle oil. The remainder is atomised in a third vessel and passed in the form of vapour with hydrogen at 460° C. over a molybdenum and zinc catalyst. This treatment converts 70 to 80 per cent. of the residuum into gasoline. The middle oil produced may be used as illuminating oil. In this manner it is possible to convert practically 100 per cent. of the raw oil without coking troubles.

If the product had been treated in one stage, however, the low-boiling constituents would have formed gaseous hydrocarbons, and the high-boiling constituents would have formed coke. R. P.

896. Process of Cracking Oil. E. W. Isom. U.S.P. 1,727,707, Sept. 10, 1929. Appl., Nov. 17, 1924.

The invention relates to a semi-continuous process in which the residue in the still is reduced to a dry dense coke, suitable as a domestic fuel or for other purposes, which can be easily removed from the still at the end of the operation, instead of reducing the charge down to pitch as is usual. The process has the advantages that a greater quantity of distillate is obtained, plugging of tar lines and coolers is avoided, and greater ease of handling coke rather than pitch.

Heavy oil is charged to a still which is not heated externally. Heat is supplied by injecting lighter hydrocarbons such as gas oil in the form of vapour, which is produced by pumping the gas oil through a coil heater, thence through perforated pipes placed in the still bottom. The heavy oil is thus rapidly heated to cracking temperature, when feeding of additional

heavy oil is begun, and continued throughout the major portion of the run. The heavy oil feed is then discontinued and the working pressure gradually reduced to promote vaporisation. As the coke approaches dryness the feed of light oil is stopped and steam introduced instead. After the coke is free from oil the fire in the coil furnace is shut off, and the steam feed is continued to cool down the apparatus.

R. P.

897. Improvements in Process for Treating Heavy Oils. C. A. Jensen for the Jenkins Petroleum Process Co. E.P. 319,040, Sept. 13, 1929. Appl. June 13, 1928.

In the continuous cracking of oils in a closed circulatory system, it has been proposed to circulate the oil within the cracking zone by means of propellers. It has now been found that the best results are obtained by driving the oil at a very high speed, and by imparting only a small increment of heat to the oil during its passage through the heating zone. When extreme rapidity of circulation is attained it is accompanied by turbulency of the oil in the cracking system, causing a uniform dispersion of solids and constant rate of gasoline formation throughout the body of oil.

The plant consists of a horizontal still connected at each end by sluiceways to transverse drums situated beneath the still. The drums are connected to each other by heating tubes and are at different levels so that the heating tubes slope upwards towards the burner end. A propeller is situated in the longer sluiceway and serves to circulate the oil from the still through the lower drum and heating tubes to the upper drum thence back to the still. The heating tubes only are subjected to the furnace heat; all other parts of the plant are outside the furnace. Simple propeller devices are liable to set up vortex effects if run to obtain increased speed of circulation, but an improved construction consisting of a flywheel rim translates vortex effect into a positive drive in the present invention.

Former practice developed a speed of 10 to 12 feet per second through the tubes, but this rate has proven insufficient to prevent a film of carbon depositing and failed to maintain a uniform dispersion of solids introduced into the oil, such as lime, to prevent corrosion. By introducing the positive balanced construction of propeller, vibration at high speed is eliminated and blacklash minimised, thus insuring full benefit from the improved apparatus. A rate of upwards of 18 feet per second through the tubes may be employed with a reduction of both speed of rotation and diameter of propeller as compared with the rimless type. This speed of circulation is found to be sufficient to prevent deposition of any solid material, and this is highly important, particularly where a comparatively heavy charge of lime is necessary to achieve the best results.

With the cleanliness of tubes produced by the increased force of circulation a maximum rate of heat exchange occurs with a corresponding reduction in fuel consumption, and coincident with the above conditions a minimum of heat is transferred per lb. of oil passing through the heating zone, thus progressively and uniformly heating the charge by rapidly recurring cycles. Such conditions result in the lowest yield of permanent gases and minimum degradation of the raw stock.

R. P.

898. Converting Heavy Mineral Oils into Lower Boiling Products. W. G. Leamon. E.P. 317,868, Aug. 14, 1929. Appl. Mar. 14, 1928.

The object of the invention is to provide a continuous cracking process without interruption due to the deposition of carbon in the cracking zone, by carefully controlled operating conditions in the vapour phase.

The cracking stock, a topped crude oil for example, is pumped through exchangers into a pipe still at a pressure of about 100 lbs. per sq. in. and heated to a temperature from 360° to 425° C.

The oil leaving the pipe still passes into an upright chamber, provided with baffles in the upper part, wherein unvaporized and tarry matter collect and may be withdrawn. The vapours pass into a centrifugal separator where any residual liquid and tarry substances are removed. The clean vapours return to the furnace, passing through a second heating coil subjected to more intense heat than the pipe still, at a high velocity, and rapidly brought to a temperature in the neighbourhood of 600° C.

In practice the vapours are at a temperature which is insufficient to cause substantial coking in the coil, and the maximum temperature should not much exceed 600° C. By the time the superheated vapours reach the outlet of the coil the pressure has dropped to about 10 lbs. The vapours leaving the coil enter the cracking unit, which may be a vertical chamber containing a mass of porous contact material such as pumice stone, "sil-o-cel" or silica gel. The cracking unit is heavily lagged to cut down heat losses. The temperature in the centre of the cracking zone may be from 530° to 550° C., or somewhat higher under normal conditions, and the temperature at the outlet about 20 to 30° C. lower. Heavy liquid products formed in the cracking chamber may be withdrawn continuously or intermittently. Substantially no carbon separates in the cracking zone, and it is feasible to run the plant continuously for weeks or even months without cleaning the chamber. The vapours finally pass through heat exchangers and dephlegmators. The reflux oil is returned for further cracking, and the overhead spirit is condensed. In the process described automatic control is provided by utilizing the variations in gas yield from a predetermined optimum to control the furnace temperature. R. P.

899. Conversion of Heavy into Light Hydrocarbon Oils. E. C. R. Marks, Assr. to Deutsche Erdöl Akt. E.P. 316,600, July 30, 1929. Appl. April 30, 1928.

The invention relates to improvements in the process and apparatus for the conversion of heavy into light oils, of a character in which the heat treatment is effected in three entirely separate zones (a heating, a cracking and a separation zone) without any substantial increase in heating in the conversion zone, and in which the latter is not used for the deposition of coke. The accurate temperature adjustment of the cracking zone is a deciding factor as regards the efficiency of the whole installation, since the speed of the cracking reaction depends to a great extent on the temperature. The heating zone is divided into adjustable sections in such a manner that from the point of lowest cracking temperature (about 390° C.) to the end of the heating zone, the supply of heat is increased. The conversion zone consists of a long, thin, serpentine tube providing a large heating surface, and thus a gentle transmission of heat to the oil. The latter is passed through the tube at a speed designed to avoid the deposition of coke. The first section of the heating zone is heated by flue gases coming from the second section and from the conversion zone, the second section being heated by means of separate burners. The flue gases are controlled by means of a valve provided in the chimney of the still. The length of the second heating section can be varied with respect to the length of conversion zone by means of valves or sliding partition walls, or by the regulation or disconnection of individual gas burners. W. H. T.

900. Rectification of the Gaseous Products of Vapour-phase Cracking. *Markowitch Neft. Khoz.*, 1929, 1, 67.

A description is given of the rectification of the gaseous products of vapour-phase cracking at the Nefte-gas Refinery, Leningrad. The plant is designed

for the production of Blau gas, and fixed gases and cracked gasoline are by-products. Cracking is carried out in Pintsch retorts of cast steel at atmospheric pressure, and at temperatures of 600–620° C. in the vapour zone. Fixed gases obtained in rectification are introduced into the reaction zone in order (*a*) to lower the partial pressure of the cracked products and increase the yield of unsaturated hydrocarbons, (*b*) to increase the concentration of hydrogen, methane and ethylene in the reaction zone and thus facilitate the formation of heavier hydrocarbons, (*c*) to condense the light saturated compounds into molecules of 3–6 carbon atoms. The cracked vapours are passed through air and water condensers, and the residue gas is scrubbed and treated in order to remove fog. The residue gas contains on an average (by vol.) 4·7 per cent. of isobutylene and higher, 2·7 per cent. of diphenyl, 19·2 per cent. of propylene, 22 per cent. of ethylene, 9·3 per cent. of hydrogen, and 42·1 per cent. of saturated hydrocarbons. The system of rectification, whereby the fixed gases and gasoline hydrocarbon are removed, is described with flow chart and figures. The gasoline hydrocarbons are compressed and, by expansion, serve as a source of cold for condensing the Blau gas fraction. The Blau gas is liquid at 100 atm. pressure and 15° C., with a mean specific gravity of 1·04 (Air = 1) and is constituted as follows (by vol):—Ethylene 22 per cent., propylene 23·2 per cent., diphenyl 3·2 per cent., isobutylene and higher 5·2 per cent., saturated hydrocarbons 42·7 per cent., and hydrogen 3·7 per cent. The following is an average composition of the gasoline hydrocarbons:—Isobutylene and higher 25 per cent., diphenyl 22 per cent., propylene and n-butylene 45 per cent., ethylene 4 per cent., and saturated hydrocarbons (Propane and butane) 2 per cent. W. H. T.

901. Ceramic Coatings in Oil Cracking. J. C. Morrell and W. F. Faragher. *Chem. and Met., Eng.*, Oct., 1929, 36, 10-596.

The increasingly large proportion of oils of high sulphur content which are used as cracking stocks have intensified the corrosion problem. Of the two general forms of corrosion in oil refinery equipment, "acid corrosion," which is exhibited mainly in the cooler parts of distillation apparatus, is due to the formation of hydrochloric and sulphuric acids. This form of corrosion is easily prevented by injection of alkaline solutions into the vapour lines from the stills. Corrosion caused by sulphur or its compounds is the most destructive and it appears that H_2S is the principal cause. H_2S is formed by thermal decomposition of different sulphur compounds found in petroleum and the extent of corrosion is not a function of the per cent. of sulphur in the cracking stocks, but apparently of the per cent. of H_2S formed in the cracking process.

The analysis of gases from the cracking of high sulphur cracking stocks shows from 7 to 13 per cent. of H_2S.

The destructive effects of H_2S at high temperatures is shown, aluminium being the most resistant whilst copper is the least. Mild steel shows a high rate of corrosion, and, as this is the usual material used in the construction of cracking plant, there is the danger of rupture of the corroded parts and economic loss due to the replacing of new elements in the cracking system.

Many methods have been suggested for overcoming the problem, such as the use of chrome steel, calorised tubes, injection of alkaline chemicals, plating or coating with resistant metals. These methods all have disadvantages and, after a careful study of the problem, it was concluded that a lining of non-metallic materials of ceramic character would be the most satisfactory and a large number of substances were tested.

A satisfactory coating should be resistant to acidic substances and gases, permanent under rapid heating and cooling, strongly adherent to the steel

surface, should not crack during initial drying, and be easily applied, *e.g.*, by spraying. It is also necessary that the coke or pitch which accumulates during the process be easily removed from the coating.

Satisfactory results were obtained with various combinations, a practical mixture was selected and a method of application and curing was devised.

The chamber to be lined is cleaned and sand blasted, then sprayed with the lining mixture composed of furnace cement 60 lbs., white silica foundry sand 30 lbs, asbestos short fibre 1½ lbs., water glass 1 gallon, water about 1½ pints (sufficient to give proper consistency). Each 100-lb. batch should cover 100 square ft. ¼ in. thick. A brushing solution is then applied, the brush being only damped or slightly wet to avoid thinning the coating. This solution is prepared by mixing 6 lbs. of furnace cement with 1 gallon of water glass and about 3 pints of water.

If the mixture is sprayed too thick it is brushed down to the desired ¼ in.; if sprayed too thin it is brushed over with fresh mixture and afterwards finished with the brushing solution. The lining is air dried for 12 to 18 hours or more, the temperature being kept steady to prevent cracking.

The curing equipment consists of a gas burner with an air jet and a sheet-metal pipe for combustion space. This is placed at the bottom of the lined chamber and the temperature gradually raised under specified conditions. The finished coating is hard and difficult to break. Some of these linings have been in use for over a year and apparently have improved with age. Tests show that it is resistant to acids and alkalis. R. P.

902. Heat Treating Hydrocarbon Oils. R. C. Osterstrom and C. R. Wagner. E.P. 317,507. Aug. 9, 1929. Appl. May 9, 1928.

The invention provides for an oil cracking system wherein conversion of relatively high boiling-point oils into oils of lower boiling point takes place while the oil is maintained in the vapour phase, the operating temperatures being so regulated as to substantially prevent the formation of carbon. A chief feature of the invention lies in the reduction to a minimum of the time during which the oil is subjected to the temperature range from approximately 600° to 1,000°F. (315-540°C.).

Oil from a "hot oil" tank is pumped through a primary heater of suitable design wherein its temperature is raised to 600°F. approx. With some types of oil temperatures of 800°F. (425°C.) may be safely attained at this stage without cracking the oil. Superheated steam is injected into the pipe line conveying the heated oil and vapours to the evaporator where the mixture is discharged below a constant liquid level. A rough separation is effected, the lighter ends distilling through a short dephlegmating column to a separator at temperatures of 600-800°F. approx.; the heavier ends collecting in the bottom, the excess above a certain level being removed from the cycle as fuel oil. The evaporator effectively traps any free carbon from the primary heater. The vapours pass horizontally through the separator, which is jacketted by hot flue gases, thus ensuring the passage of nothing but dry vapours and steam to the converter; any condensate is trapped by vertical baffle vanes, and is returned to the hot oil tank.

The converter may be of several types. One type resembles a fired pipe still; the first passes are arranged in the hottest part of the furnace, partly in the radiant section, whereby a rapid and substantially instantaneous rise of temperature takes place from 600° or 800° to 1,000°F. or over, at which latter temperature the conversion is completed. The converter elements are preferably provided with cores of a catalytic material having the properties of the oxide of a metal, such as iron or nickel, which is susceptible to two or more stages of oxidation. The catalytic cores are of less diameter

than the internal diameter of the converter tubes, being designed to leave an annular space for vapour flow. By tapering the core slightly to a lesser diameter at the outlet end of the element, the velocity of vapour flow may be regulated as desired. Another type of converter employs a single elongated tube of larger diameter, containing a tapered catalytic core as before, heated by an electric furnace. This type simplifies many mechanical difficulties.

The velocity of vapours entering the converter is 2,000 ft. per min. approx., while at the exit velocities of 5,000 or even 7,000 ft. per min. may be attained, and the time during which the oil vapour is subjected to conversion temperatures is less than two seconds. This secures the desired conversion without over exposure, thereby eliminating the formation of excessive fixed gas or free carbon. Pressures not exceeding 30 lb. per sq. in. may be employed.

Vapours leaving the converter pass down one side of a cooler or "tar leg," in intimate contact with a finely divided spray of raw make-up oil, which secures a substantially instantaneous drop in temperature from above 1,000°F. to a critical temperature below 600°F. approx. Uncondensed vapours are led from the other side of the tar leg to suitable fractionating equipment, where the heavier portions are rejected as liquid and returned to the bottom of the tar leg, from which they are withdrawn to the hot oil tank as recycle stock together with the pre-heated make-up oil. A liquid level controller returns excess oil from the hot oil tank to the source of supply. Vapours of the desired end point are taken off the fractionating equipment, condensed, and led to a decanter where separation of gas, oil, and water is effected.

When necessary under certain conditions, vapour flow through the converter can be arrested, and superheated steam circulated, thereby removing carbon deposit; this purging may also be effected by air. L. H. A.

903. Modern Cracking Installation. *Pet. Times*, 1929, 22, 160.

The cracking stock is gas oil and the gasoline yield is about 62 per cent. Units run from 1,200 to 1,500 hours without shut down. This is possible by using three reaction vessels for two units so that two are in use while the third is being cleaned. Three products are made: a gasoline of high anti-knock properties, a furnace distillate, and a viscous low cold test fuel oil. The gasoline is low in gum content for a cracked spirit and has a boiling range of from 88° to 415°F.

An elaborate system of heat exchange is used and condensation of the products is mainly effected in this system. Fractionation is effected by two bubble towers connected in series.

The furnace runs at about 1,000 lbs. pressure, the reaction chamber at about 650 lbs., and oil leaving here is reduced to 65 lbs. pressure. There is a very complete system of automatic control by regulating devices located at a convenient central point. W. L.

904. Some Aspects of Cracking. *Petr. Times*, xxii, 558, 544.

The article is a review of a book just published in New York by C. Dewitt Hurd entitled "Pyrolysis of Carbon Compounds." The volume is largely a compilation of the knowledge accumulated in connection with the subject and contains much information of value to the technologist, but references to the oil cracking industry can be considered of general interest. A feature of this section is a discussion of the vapour phase cracking processes which have lately become prominent as a source of anti-knock fuel. Vapour phase processes can be worked at low pressure, hence the cost of plant is

less than in pressure distillation processes. The excessive formation of permanent gases is probably the main drawback in vapour phase cracking. In pressure distillation processes there is, for each oil, a relationship between temperature and pressure, whereas in vapour phase processes the conditions of temperature and pressure are independent. Vapour phase processes are fundamentally similar. A stream of oil is raised to cracking temperature in a heating zone and the gasoline formed remains with the uncracked oil during its passage through the furnace. This results in exposure of the gasoline to cracking conditions for a considerable time after its formation, during which some recracking can occur with formation of permanent gas. The production of aromatic hydrocarbons requires higher temperature than ordinary cracking for gasoline production. Temperatures from 600° to 1000° C. are used in the Rittman process at pressures of at least 6 atm.

R. P.

905. Material for High-Pressure High-Temperature Oil Cracking Plants. *Petr. Times*, xxii, 558, 562.

The great developments in the field of high-pressure steam have created demands for materials with a high degree of strength at the temperatures employed. In exceptional cases the pressure developed may be as high as 1800 lb. per sq. in., with correspondingly high temperatures, which occasions severe demands upon the materials of construction. Another feature is the high cost of such materials. Under oil cracking conditions the refiner is also faced with the question of corrosion due to sulphur and hydrochloric acid contained in the cracking stock.

A new cracking plant of the tank and tube type designed to crack 3000 barrels daily has recently been installed by the Standard Oil Company of Ohio, and is said to produce approximately 70 per cent. of gasoline. The plant consists of three soaking drums 58 in. inside diameter by 40 ft. long forged from solid steel billets with walls 5½ in. thick. The two furnaces have radiant, convection, and reboiler sections. The cracking tubes in the radiant section number 52 of seamless chromium nickel steel 4½ in. outside diameter, ¾ in. thick. These tubes are exposed to the highest temperature of the furnace, 1625° F. on an average.

The tubes of the convection section are 35 in number, 4 in. outside diameter, made of carbon steel with ¾ in. walls and fitted with gill rings, also 13 tubes without gill rings. The reboiler section consists of 40 tubes 4 in. outside diameter, ¾ in. thick, and below the reboiler are 16 superheater tubes 2 in. diameter for superheating steam.

All tubes are 28 ft. long, making the total length of tubing more than five-eighths of a mile. The header boxes connecting the tubes are of extra heavy forged steel.

The strength of the entire piping system is in proportion. Heavy seamless tube is used throughout and all valves are of forged steel. All fittings are tested to 1350 lb. pressure. All joints are ringed joints using a soft steel ring which fits tightly into a groove. Low chromium steel bolts are used which have a tensile strength of 130,000 to the sq. in. and an elastic limit of 110,000 per sq. in.

R. P.

906. Research on Cracked Residues and Cracked Oil Distillates. A. Sachanen and M. Bestuschew. *Petr. Zeit.*, 1929, **25**, (1307-09).

The physical properties of cracked oil residues are described, *viz.*, specific gravity, viscosity, flash point. Balachany-Masut, of sp. gr. 0·908 at 15°, $E_{50} = 6.17$, setting point −20° C., flash point (open) 161° C., was utilised as the initial substance, and cracked at 425° under 15 atm. pressure for

32 mins., 1 hr. 32 mins., 3 hrs. 2 mins., and 6 hrs. 2 mins. respectively. The cracked residue is that which remains after distillation of the benzine and kerosene fractions to 300° under normal pressure. This was distilled in a high vacuum under 6–15 mm. mercury pressure, and the boiling points at normal pressure calculated from Ramsay and Young's formula. The results of this work are given in tables for the various cracking periods. It is shown that the cracked oil distillates differ greatly from the oil distillates of the initial substance, and this difference increases for longer periods of cracking. The paraffins are slowly decomposed by cracking, and undecomposed paraffins are concentrated in the cracked residues. The cracked oil distillates thus have a higher setting point after longer cracking. From molecular weight determinations and elementary analyses of some cracked oil distillates it has been shown that the hydrocarbons contain chiefly aromatic and hydro-aromatic hydrocarbons. The fraction boiling between 280° and 354° with 3 hrs. 2 mins., cracking contains isoamylnaphthalene and heavy aromatic hydrocarbons. The solid product from fraction 2 with 6 hrs. 2 mins. cracking is identical with m-dimethylanthracene. Dehydrogenation as well as decomposition are brought about by cracking. W. S. C.

907. Combined Distilling, Cracking and Gas Making. A Schwarz. U.S.P. 1,733,135. Oct. 29, 1929. Appl. Jan. 5, 1927.

A method for complete and continuous conversion of heavy oils into light oil and gas without the deposition of residues and without undesirable by-products is claimed. The process is designed to operate under the principle of counter flow. The oil first encounters heat from the flue gases discharged from the system and light distillates are removed. The oil then encounters progressively higher temperatures passing through a cracking operation yielding products at different points and increasing the content of carbon in the residue till towards the high temperature end fixed carbon segregates. During the progress of this treatment water is converted into steam and superheated high enough to enable its oxygen to combine with the fixed carbon when they are brought together, thus gasifying the residue and producing a mixture of oil and water gas.

The treatment proceeds at maximum efficiency of the fuel employed, as the heat at its maximum intensity is used to produce gas, and heat is gradually abstracted from the flue gases by operations which are proportioned to cause efficient heat exchange between the heating gases and the material under treatment.

The plant consists of a firebox from which hot gases pass into a series of heating chambers. In the first chamber vertical retorts of chrome nickel steel are situated and in which the residue is converted into gas. The second chamber contains a bank of tubes and a vaporising drum, and the third chamber has a steam superheater. The gases then pass through the fire tubes of a water boiler into another chamber wherein the feed waterheater and oil preheater are placed, and from which the flue gases escape to the stack. The crude oil is pumped through the preheater and then passes into the vaporising chamber, from which it is circulated through the bank of tubes back to the evaporator by a circulating pump. The light oil vapours from the evaporator are condensed and run down. A branch line from the circulating pump leads to the retorts and lines take the gas from the retorts through gas scrubbers to the gas holder.

Water is pumped through the heater to the boiler. Steam from the boiler is taken into the superheater and passes to the retorts, where it enters with the oil, thereby spraying the latter. An oil and steam nozzle sprays the oil at a tangent against the wall of the retort, causing the stream to

travel in a spiral along the inside wall. The gas line extends two-thirds of the way down inside the retort in order to prevent undigested oil from passing out.

With a mid-continent oil the temperature of the oil preheater was 200° F., whilst the water heater temperature was 180° F. and the boiler was maintained at 80 lb. pressure. The superheater temperature was from 1200° to 1400° F. Ten gallons of oil produced 1000 cu. ft. of gas from which 2 gall. of gasoline and 3 gall. of gas oil were obtained, leaving only 5 gall. to enter the retorts for gas production. R. P.

908. Apparatus for Cracking Oils. S. Seelig. U.S.P. 1,732,664. Oct. 22, 1929. Appl., Dec. 29, 1926.

In continuous cracking of oils by conducting them into a bath of molten metal and allowing them to ascend through the fluid, it is difficult in practice to maintain the correct temperatures constantly and accurately. Several arrangements have been proposed operating mechanically by regulating the flow of oils or the molten metal, but experience has shown that such devices operate imperfectly.

In the present invention a cylindrical vessel containing molten metal and packing rings is externally heated in a furnace. The vessel is also fitted with a spiral tube which forms an annular space between the inner wall of the vessel and the spiral tube. The object of this is to control the temperature of fluid within the reaction chamber by circulating a gas, water vapour or the oil feed through the spiral.

When the apparatus is operating the spiral forms an insulating wall which only slowly takes up the temperature of the outer wall, and thereby balances changes of temperature. The effect of conducting the oil to the bottom of the vessel is to cause an upward flow of the fluid contents, including the molten metal, and the spiral tube is designed so that the molten metal will flow outwards towards the shell of the vessel and return to the bottom by the annular space. R. P.

909. Distilling and Cracking Oils and the like by Contact with Salt and Metal Melts. T. Seifer, B.P., 319,820. Sept. 30, 1929. Appl., June 28, 1928.

A process and apparatus is described for distilling and cracking oils and the like by contact with salt or metal melts, in which the melt is constrained to circulate between treating and heating chambers, by delivering the body to be treated at a high pressure to a spraying nozzle placed in the melt so that the body is projected in the direction in which the melt is to circulate. The heating and treating chambers are placed one below the other, or side by side, or in concentric relation. The circulation is augmented by an auxiliary pressure medium. The feeding device consists of an inner hollow cylinder formed in two parts, and its end in the autoclave is made of a naturally hard refractory substance, which is fitted into the annular recess in the other part of the cylinder, with packing, or it may be a vertical feed pipe fitted in the bottom of the autoclave. The body is carried from the cover plate close to the bottom, where the direction of flow is reversed by a U-bend in the feed pipe. W. S. C.

910. Process and Apparatus for Distilling and Cracking or Decomposing Bodies such as Oils and the like by Contact with Salt or Metal Melts. T. Seifer. B.P. 320,126. Sept. 30, 1929. Appl., June 28, 1928.

A process and apparatus are described for distilling and cracking oils and the like by contact with salt or metal melts, in which a thermodynamic circu-

lation of the melt is promoted between a treating chamber and a heating chamber connected by a downwardly directed pipe, or a horizontally disposed heating chamber and a treating chamber arranged vertically. In the latter case, the treating chamber is connected by a downwardly directed pipe, the two chambers meet outside the furnace which heats the heating chamber, and the liquid to be treated is supplied to the treating chamber at the bottom.

W. S. C.

911. Converting High Boiling Hydrocarbons into Low Boiling Hydrocarbons. H. Terrisse and L. Dufour. E.P. 305,981. Oct. 31, 1929. Appl., Jan. 21, 1929.

High boiling-point oils are cracked by heating in the presence of hydrogen, water gas or the like, under a pressure of 5 to 40 kilograms per square c.m. Ferrous chloride is added as a catalyst and also acts as a desulphuriser. The reaction is accelerated by the addition of active carbon, brown coal, lignin or peat.

W. L.

912. Method of Distillation (Cracking). C. P. Tolman. U.S.P. 1,733,143. Oct. 29, 1929. Appl., Nov. 22, 1924.

The purpose of the invention is to provide an efficient method of transferring heat to the material without local overheating such as is produced when oils are cracked in pipes and stills. This is accomplished by using a medium which will not mix with the oil and which will become liquid at or above the final temperature. In the process described mercury is used as the medium for heat transfer. A mercury boiler supplies mercury vapour to the upper part of a vertically disposed cracking chamber. Raw oil is sprayed above the mercury vapour inlet and a pump circulates liquid from the bottom of the cracking vessel through another spray in the roof of the chamber. A vapour pipe connects the cracking vessel to a condenser, and the condensed mercury and oil flow to a separator whence the mercury is returned to the boiler, and the oil passes out to its receiver. The method may also be used for treating solids such as oil shale, coal or lignite, by providing charging and discharging doors in the reaction chamber.

It is claimed for the method that the material undergoing cracking is subjected to a very even temperature, which is of great importance; also the presence of large quantities of metallic vapour at a comparatively high temperature produces a catalytic effect, promoting the decomposition of heavy into light oils.

R. P.

Oilfield Technology.

913. Borehole Clinometers. Armenæ Chahnazaroff of Baku. E.P. 320,218. Oct. 10, 1929. Appl. Sept. 24, 1928.

The device consists of a casing, containing recording apparatus, which can be lowered down the bore-hole to measure and record the deviation from the vertical of the hole. This outer casing is provided with rollers, pressed against the bore-hole walls by springs, to facilitate upward and downward movement, to prevent turning in the bore-hole and to ensure the casing remaining co-axial with the bore-hole. Enclosed in this fluid-tight casing is another cylinder, seated on cork or other yielding material to protect the instrument from shock or vibration. By an arrangement of gimbal rings in this cylinder a pendulum is always hanging vertically; the inner gimbal ring carries a clockwork device operating two pointers at right

angles. These trace curves on the paper affixed to the inside of the inner casing, so recording the angular deflection between the casing and the pendulum.

J. F.

914. Properties of Producing Oil Sands. R. C. Beekstrom. *Oil and Gas J.*, 1929, **28** (20), 70, 328.

An oil reservoir consists of consolidated or unconsolidated sands containing one, two or three of the fluids, oil, gas and water. Water-deposited sand presents the best conditions from oil accumulation and the porosity is dependent on the grain size and on the amount of cementing material and silt present.

The oil removed by natural means is that stored in the spaces between the grains of the sand. The greater part of the oil in the sand is adsorbed by the grains and cannot be recovered by natural methods. The oil is also held in the reservoir by its surface tension. Four important factors control the migration of the oil, its nature, its temperature, its capillary attraction, and its adsorptive value.

All the factors concerning oil sands must be understood and controlled before the maximum quantity of oil can be produced profitably.

Laboratory experiments have been carried out and demonstrated that by compaction alone oil migrates from one structure to another. Experiments were also made by using the different recovery methods and up to about 90 per cent. of the oil originally present was recovered.

G. S.

915. Corrosion in Big Pipe-Line Problem. L. G. E. Bignell. *Oil and and Gas J.*, 1929, **28** (15), 35, 377-378, 381.

So far research on pipe materials has not produced a satisfactory material, but it has been proved that impurities shorten the life of metals and under corrosion conditions will set up electrolytic action, especially in the presence of an acid electrolyte. Some advantage may be gained by inserting insulators, in the form of gaskets, in the pipe-line and coating the threads and couplings with insulating material.

Various coatings of the thick, thin, or plastic types are available and it may be advisable to use one or more types on one pipe-line to counteract several types of corrosion. For the protection of the coatings, a wrapping of rag paper or an asbestos mixture is used, but it is suggested that a coating be applied over the wrapping to prevent any tendency of the wrapping to pick up moisture.

Some companies are preparing "soil maps" on which the corrosive properties of the soil are noted. The information enables them to predict with some certainty the behaviour of pipe buried in a given soil.

G. S.

916. Practice in Handling Flowing Wells. R. W. Bond, D. L. Trax, C. D. Watson and M. Walker. *Oil and Gas J.*, 1929, **28** (20), 112, 342.

In the mid-Continent area it was the practice, until recently, to prolong natural flow as long as possible by agitation, then swab, and finally pump. Agitation and swabbing has been superseded by gas lift. Most of the wells are finished 6¼-in., 6⅝-in. or 8¼-in. casing and connected to a separator by a 6⅝-in. flow line.

The majority of the wells are put on the lift as soon as possible, the flow being up the annulus between tubing and casing, and the intermittent method

of flowing is gaining favour. The relative overall efficiency can be calculated from the formula :—

$$\text{Efficiency} = \frac{\text{actual gas-fluid ratio}}{\dfrac{0{\cdot}072\ Lg.}{\log 10 \dfrac{P_1}{P_2}}}$$

where L = Tubing depth plus trap heighth.

G = Sp. gr. of fluid.

P_1 = Approx. gauge pressure at top plus 14·7 lb. plus weight of gas minus friction.

P_2 = Abs. trap pressure.

Data on a considerable number of wells were considered, but the numerous variables and flow string combinations do not allow of definite conclusions being drawn at present. G. S.

917. Deep Wells Create Casing Problems. L. G. E. Bignell. *Oil and Gas J.*, 1929, **28** (20), 69, 362-364.

The drilling of wells to depths of around 9,000 ft. brings new problems to the oil industry. The transition from the older and more crude tools to modern equipment has been very gradual, but has latterly required the skill of the metallurgist and the chemist together with the knowledge of the mechanical and petroleum engineer.

In one well in Logan County a string of 6⅝-in., 26 lb. casing 6250 ft. in length and weighing 162,500 lb. was successfully set. This pipe was 45-55 carbon, 0·8 per cent. manganese, yield point 55,000 to 75,000 lb., and tensile strength about 110,000 lb. This is stronger than A.P.I Grade C., which has manganese 0.35 to 1·50 per cent., yield point 45,000 lb., and tensile strength 75,000 lb.

In Oklahoma City Field it is customary to carry the 9-in. casing to about 5500 ft. Such a string of 40-lb. pipe would weigh about 220,000 lb., but as it is usually floated in the actual load on the derrick is only about 37,757 lb. This string is usually cemented, in some cases as much as 1000 sacks of cement being used.

The pipe companies have developed a special alloy, 55½ lb., 10¾-in. O.D. casing and for a 10,000-ft. string, a 4½-in. O.D. casing, tensile strength 110,000 lb., and weight about 16⅔ lb. per ft. has been developed. This pipe is rated for 9635 ft. with a safety factor of 2 to 1. An alternative 4½-in. string for 8400 ft. has a tensile strength of 95,000 lb.

Collapsing pressures are usually estimated by multiplying the length of the string by 0·5 but collapsing pressures are materially reduced by the practice of cementing behind the casing, as in the Oklahoma City Field.

Lengthening of the threaded portion is suggested to add to the strength and opinion is divided on the merits of 8- or 10-thread casing.

The practice of carrying the 9-in. casing to the top of the producing horizon should make it possible to control gas in the upper strata. G. S.

918. Use of Hydraulic Cement in Oil Wells. J. B. Case. *Petr. Times*, 1929, **22**, 156, 222.

Data relative to the sources of water, or the segregation of high and low pressure portions of zones, must be obtained for intelligent procedure, and this is best secured by co-operation between interested companies and various departments of each company.

While the operation of cementing is mainly mechanical, records show how much more efficient the results are under technical supervision. The failure of a cementing job does not eliminate the necessity for cementing, and the job should be repeated until it is successful.

General opinion holds that cement should be used as soon as possible after it is ground. The time interval for cement to pass from the fresh state to the old state varies according to conditions. Cement should be stored in a cool dry place, and sacks should not be piled on one another as pressure, due to weight, is injurious to its setting quality. When cementing the consistency of the slurry going into the well should be kept as constant as possible. When a well is wet and recementing is contemplated, often insufficient time is spent in trying to establish circulation. Instances are known where good circulation was established only after pressure had been kept on the wells for four or five days. In recementing water strings any open hole below the shoe should be plugged to within two or three feet of the shoe before recementing begins. No accelerator should be added to cement for recementing.

There are three methods of recementing water strings when no oil string or liner is present.

1. Recement through casing if free and open circulation can be established round the shoe of the casing, either to the surface or into the formations.

2. Dump cement on the bottom using a positive dump bailer, which holds sufficient cement, so that but one dump is necessary, and then apply pressure by means of a packing head and high pressure pumps. This method is only applicable when the leak is small and the water under low head.

3. Recementing through tubing under pressure. There is no occasion for cementing tubing in a well if reasonable precautions are taken. The valve on the packing head must be closed before any cement leaves the tubing. Other causes of cemented tubing are, too much cement, the use of an accelerator, and withdrawal of insufficient tubing when large quantities of cement are placed in the bottom.

The use of a cement retainer is advocated as it assures that cement is forced into position as it reaches the bottom, and that as long as cement is passing out of the tubing it is going into its intended position. Also it allows a greater pressure to be applied with safety, and prevents the return of cement into the casing when pressure on the tubing is released. The main objection to its use is the difficulty sometimes experienced in unscrewing the tubing from it. W. L.

919. Retarding Decline of Rock Pressures. S. Coleman, H. D. Wilde and T. W. Moore. *Oil and Gas. J.*, 1929, **28** (20), 192c–192d.

The decline of rock pressure can be retarded by two methods. One is the control production, so that the gas-oil ratio is lower, and the other is to return to the oil-bearing strata all or some of the gas brought to the surface.

The mathematical relation between gas wasted and average rock pressure after removal of a portion of the oil is discussed. It is assumed that there is no water drive, and that gas solubility in the oil follows Henry's Law. The terms and symbols used are :

Gas-free oil = dead oil, *i.e.*, oil saturated with gas at 1 atmosphere.
Original conditions = pressure, temperature, sand saturation, etc., existing prior to drilling the first well.
Volume of reservoir = total volume of all pore space in the sand.
X = gas-free oil removed to a given time. The gas-free oil originally in the sand = 1.
P = original rock pressure in atmospheres.
p = mean rock pressure in atmospheres after removal of X oil.
k = constant for Henry's Law. Gas dissolved = kp.
a = coefficient of volume expansion for solution of one unit of gas in one unit of gas-free oil.

γ = fraction of total gas wasted in producing X volumes of oil.
m = ratio of volume in reservoir containing free gas at pressure P to volume containing oil.
g = mean gas wasted per unit of gas-free oil produced divided by total gas in reservoir per unit of oil under original conditions.

Volumes of gas-free oil are in cu. ft., and volumes of gas in cu. ft. occupied at 1 atmosphere and reservoir temperature. It is assumed, for simplicity, that the reservoir originally contains 1 cu. ft. of gas-free oil.

At the start the volume of gas-free oil = 1; of the liquid $(1+akP)$; of the free gas $m(1+akP)$; the total volume of the reservoir being the sum of the last two.

After X gas-free oil has been removed the gas-free oil remaining is $(1-X)$; volume of liquid remaining is $(1-X)(1+akP)$; volume of free-gas space in the reservoir

$$[(1+akP)+m(1+akP)]-(1-X)(1+akP) = X(1+akp)+ak(P+Pm-p)+m.$$

The volume of gas remaining in the reservoir $= (1-X)kp+p[(1+akp)X+ak(P+Pm-p)+m]$. This may also be expressed as $(1-\gamma)[(1+akP)Pm+kP]$.

Equating these two expressions we have:

$$(1-X)kp+p[(1+kp)X+ak(P+Pm-p)+m] = (1-\gamma)[(1+akP)m+kP] = (1-gX)[(1+akP)Pm+kP)$$

For a given field a, k, m and P are fixed, and with g set at any desired value the only variables are p and X. It is simpler to express X in terms of p, as

$$X = \frac{m(P+akP^2-akpP-p)+k(P-p-apP+ap^2)}{m(gP+gakP^2)+(1-k)p+akp^2+gkP}$$

Where all gas is held in solution m is equal to zero and the equation becomes:—

$$X = \frac{k(P-p-apP+ap^2)}{(1-k)p+akp^2+gkP}$$

Curves can be drawn to show the relation between gas-oil ratio and rate of rock-pressure decline.

It is estimated that where the gas-oil ratio has been between 1200 to 2000 cu. ft./bbl., the maximum recovery is limited to 16 to 20 per cent. The gas-oil ratio should be kept as low as possible. At 480 cu. ft./bbl. the recovery limit is 50 per cent., and at 720 cu. ft./bbl. 33 per cent.

The limit at which the return of gas becomes uneconomic is probably between 60 and 80 per cent. recovery.

By the use of one of the curves produced, a knowledge of the volume of oil in a given reservoir can be obtained. G. S.

920. Data Relating to Boring in Great Britain. J. A. Howe. *J. Inst. Petr. Techn.*, 1929, **15** (76), 557–562.

Practically all drilling in Great Britain has been in search of coal or water, except for a limited amount of boring for oil between 1918 and 1922.

Prior to 1918 the Government had no control over borings, but the Geological Survey collected all the information obtainable, much of which was made public in the form of memoirs, sections, etc.

The passing of the Petroleum (Production) Act, 1918, gave the Geological Survey the right to examine plans and sections, boring journals, cores and specimens collected.

The Mining Industry Act, 1926, gave facilities to the Department of Scientific and Industrial Research in respect of boreholes sunk in search of minerals, and the duty of collecting and recording geological data was delegated to the Geological Survey.

Fossils and rock samples are examined by palæontologists and geologists, and graphic sections are prepared on a scale of 40 or 100 ft. to 1 in.

The notification of water wells is not compulsory, but the Survey continues its useful work in this direction by close co-operation with the firms engaged in this work. G. S.

921. Some Misconceptions of Soil Corrosion. K. H. Logan. *Oil and Gas J.*, 1929, **28** (15), T64.

The corrosion of buried metals is affected by temperature and rainfall, characteristics of climate rather than of soil. Few soils have definite physical and chemical characteristics in the sense that a metal or chemical compound has, consequently, soil corrosiveness cannot be expressed with accuracy.

The electrolytic theory was once accounted for by differences in surface conditions of the metal, but later observations show that potential differences also result from conditions external to the metal. It is also concluded that corrosion cannot be eliminated solely by the use of a pure metal.

Tests have shown that the corrosion rate of cast iron does not differ greatly from that of other ferrous pipe materials, and may be rather rapid in some soils.

The belief that soil corrosion and stray-current corrosion can be distinguished by the distribution of the products of corrosion is incorrect. Localised pitting, a characteristic of electrolysis, may also be caused by a localisation of bad soil. Graphitic corrosion may also be equally caused by electrolysis or by corrosive soils in which the oxygen supply is limited. G. S.

922. Metallurgy of Hard-faced Rotary Bits. W. A. Wissler and C. W. Metzger. *Oil and Gas J.*, 1929, 28 (16), 75, 146.

The discussion is divided into three sections, the first dealing with the steel used for the body of the bit, the second with the welded junction between the steel and the hard-facing alloy and the third with the alloys used for the hard-faced cutting edge. With regard to the steel for the bit, forging should be carried out at a specific temperature range and should, if necessary, be reheated rather than heated too high at the start. The junction of the hard-facing metal and the steel base may be made either by oxy-acetylene or metallic arc welding, but generally the former is preferable. This type of welding allows the metal built up on the cutting edge to be shaped much more accurately so that less metal is required, also a more uniform deposit may be obtained with oxy-acetylene welding. Determination of the red hardness, that is, of the hardness at a red heat, of different alloys will show their comparative value as wear-resisting materials. The most suitable alloy for hard facing is the one having the greatest hardness while red hot. Non-ferrous alloys of the cobalt-chromium-tungsten type have greater red hardness than any other known hard facing metals. Diamond substitutes consist mainly of tungsten carbide. The best product is a compromise based on sacrificing some of the possible hardness to secure the necessary strength and toughness and the value of the product must be estimated on the basis of these two properties. W. L.

923. Causes of Sucker Rod Troubles Sought. W. C. Shutts and B. L. Moore. *Oil and Gas J.*, 1929, 28 (16), 38, 130.

This paper gives the results of experiments made in order to determine the cause of sucker-rod trouble, the effects of counter-balancing and the overall power efficiency of pumping equipment. The weighing of wells has played an important part in this analysis.

A recording dynamometer is described, which was used to obtain indicator cards showing the weight on the polished rod and its travel. These cards, together with power readings taken at the motor, make it possible to determine the most efficient pumping motion, the effective length of plunger stroke, the correct weight of counter-balance, the effects of counter-balancing and the vibratory stresses that are set up in a string of rods by irregular pumping motion.

The indicator cards obtained suggest the possibility of the existence of a critical speed at which a pumping well might be operated and a more efficient plunger movement obtained than at either an immediately higher or lower speed.

Less differential in rod stress will increase sucker rod life and allow of more efficient counterbalancing. Considering a pumping well as a machine it can be balanced, not considering certain factors such as friction, acceleration, gas, etc., as follows :—

Up-stroke of the rods : $Bw+Wp=Fw+Rw \quad Wp=Fw+Rw-Bw.$

Down-stroke of the rods : $Bw-Wp=Rw-Wb \quad Wp=Bw-Wb+Rw.$

In a balanced machine Wp will be equal on the up and the down-stroke and so

$$Bw=Rw+\frac{Fw-Wb.}{2.}$$

where Bw = Weight of counterbalance
Wp = Maximum force at crank end of beam
Fw = Fluid load supported by valves.
Rw = Weight of rods
Wb = Buoyant effect of fluid on rods.

W. L.

924. Superhard Metals for Tool Facing. H. J. Morgan. *Oil and Gas J.*, 1929, **28** (20), 176, 178.

Under ordinary conditions the changing of a rotary bit takes from 2½ hrs. for the round trip at 4000 ft. to 5 hrs. at 7000 ft. To reduce time losses hard-facing metals are welded on to the cutting edges of the bits to afford protection from abrasive wear.

A superhard metal has been developed with a hardness of over 9 in Mohs scale, i.e., it will scratch a sapphire or ruby.

The most important of the hard substances are the compounds containing carbon, silicon and boron. In order to obtain the greatest hardness and resistance to abrasion, it is important to get the right amount of carbon and the correct crystalline structure.

There are three main classes of tungsten carbide products : (1) In the wrought state, as Carboloy, Dymondite, Wedia, etc., (2) in the cast state, as borium, dymonettes, titan, etc., and (3) corresponding to casehardening, an example of which is Blackor.

The Carboloy type has not been extensively used on drilling bits on account of its high cost. The second type, the insert metals, are in general use in California. Blackor is a granular material, 1 oz. of which covers five sq. in. It can be melted by the electric arc to form a solid cutting face of any shape and hardness. Three or more layers give a hardness of over 9. G. S.

925. Selection of Motors for Pipe Lines. L. J. Murphy. *Oil and Gas J.*, 1929, **28** (15), T-206-210.

The choice of electrical equipment for pumping should be that which will give the lowest cost per barrel over a period of years and under all probable conditions of loading. Owing to low cost the proportion of centrifugal pumps

is increasing, and the article is limited to the application of motors to this type. Any type of motor can be applied to the centrifugal pump, the main requirement being that it shall have sufficient torque at any speed to overcome the torque required by the pump at that speed. The squirrel cage motor is the least expensive, simplest and most rugged type. The power factor is fairly high. It has no wearing parts other than the two shaft bearings, and fire hazard is at a minimum owing to absence of brushes or collector rings. The synchronous motor, when used for pipe-line pumping, is usually installed with a direct connected exciter for each motor. The power factor is usually 100 per cent. because the station is rarely in the vicinity of any low factor load for which a correction could be furnished, although in special cases a correction can be usefully applied. The synchronous motor is about 2 per cent. more efficient than the squirrel cage type, but its initial cost is about 40 per cent. higher. Wound rotor motors have not been utilised for driving centrifugal pumps, and it is unlikely that they will be owing to the difficulty of operating the line at reduced capacity. Other factors are frequently more important than motor characteristics. These include the percentage of time at which the pump will operate at reduced capacity, the nature of the power supply contract, etc.

When a centrifugal pump is operated below its normal capacity, either by throttling or working at a reduced pressure, the efficiency falls appreciably. If the speed can be reduced the loss is not so great.

Unfortunately, there are no inexpensive and reliable methods of reducing the speed of alternating current motors. The use of D.C. with a motor generator set is too expensive, and the overall losses make the all-round performance poor. The slip ring motor may be run at reduced speed by means of a secondary resistance, but the overall efficiency makes this method less economical than the use of a series of squirrel cage or synchronous motors arranged so that some of the units in a line may be shut down at times of low demand. An alternative is to run at full output for a part of the day and then to shut down. The economy of this method depends on the nature of the power contract. Probably the best solution is to instal either a two-speed motor, or, better, two separate motors, one being designed to run at two speeds. The lower speed should be suitable for minimum output and the higher for maximum.

R. S.

926. Draw Works Drum for use in Well-boring and like Plant. National Supply Company, Delaware, U.S.A. E.P. 299,006, Sept. 23, 1929. Appl. June 22, 1928.

The draw works drum used for well-boring and like plant is made in half sections, the adjacent ends of these being provided with outwardly extending flanges finished on the inside faces to fit together, and on the outside faces to fit into a channel in a split locking ring. This locking ring, finished to fit the flanges on the outside faces and peripheries, but not the channels extending along the drum from these flanges, is sprung over the flanges and secured to the half sections by annular welds spaced longitudinally.

The inwardly extending flanges at the adjacent ends of the half sections are made a loose fit on the shaft to allow the drum to yield slightly at the centre. Webs spaced round the inner periphery join the flanges at either end of the half sections, so preventing the flanges at the outer ends from spreading away from the drum barrel. The drum may be keyed on to the shaft which supports it, or revolve round this shaft, being driven from an independent shaft.

J. F.

927. Repressuring in Depleted Oil Zones. C. M. Nickerson. *Oil and Gas J.*, 1929, **28** (20), 184, 186.

A depleted lease with a production as low as 3 to 5 bbls. per well per day can be repressured, and by injecting gas in an area as small as a quarter section the benefits may be confined to the property.

The usual compressor in the natural-gas gasoline plant in the lease has sufficient capacity to deal with the volumes and pressures necessary. Intermittent gas injection eliminates blowing excess gas to air.

The benefits of repressuring include increase in production of offsetting wells, decrease in water production, increase in wet gas production, and an increase in gasoline from the wet gas.

When the injection well is returned to production its daily yield will generally be in excess of that before gas injection.

Increased back pressure may assist the control of the direction of gas migration, and the longer the distance travelled by the gas underground, the greater the benefit obtained. G. S.

928. Method of Tubing High Pressure Wells. H. C. Otis. *Oil and Gas J.*, 1929, **28** (20), 169, 172.

A better gas-oil ratio can be secured by flowing a well through tubing of the correct diameter, and the equipment described permits wells to be correctly tubed, after bringing in, with no danger of losing control, fire, or loss of production. The equipment also permits the tubing to be plugged, snubbed out, and pipe of a different diameter snubbed in without waste or danger.

Two types of control head are general. One has a gas- or fluid-tight seal round the pipe and maintained by a rubber ring held by the pressure in the well. The other type uses steel rams, faced with packing, and held by manual mechanisms against the pipe. Two of these latter heads are necessary to permit the passage of couplings.

The snubbing equipment has two principal members, the travelling and the stationary snubbers. The former force the pipe in against the well pressure, and the latter prevent it from being blown out while the travelling snubbers are being moved. When sufficient pipe has been inserted to allow of its falling by its own weight, the snubbing equipment is easily removed.

In order to close the bottom of the tubing, to prevent flow of oil or gas, a method of breaking a cast-iron disc from the underside has been worked out. G. S.

929. Impact Tools. Pennsylvania Gasoline Drill Company, Philadelphia, U.S.A. E.P. 293,856, Sept. 16, 1929. Appl. June 15, 1928.

This invention is of the type of percussion tool for which C. S. Somervell was granted an E.P. 219,202, Sept. 3, 1923, but is improved and its construction simplified to provide greater efficiency and durability.

The actuating piston reciprocates in an air-cooled cylinder of the single-acting two-stroke internal combustion engine type.

The body is in three sections, each of chrome nickel steel forgings, heat treated to withstand heat and shocks to which each is subjected. These sections are an explosion cylinder section, a rotor cylinder section, and an impact cylinder section. The whole is assembled with bolts whose axes are parallel to the axis of the cylinder, to eliminate shearing stresses, spigotted joints being employed to give correct alignment, so that either end section can be removed without disturbing the remaining two.

The actuating piston, reciprocating in the explosion cylinder, is connected by a crosshead running in hardened slots in the rotor cylinder to a double

piston in the impact cylinder. By an arrangement of rollers on the cross-head arms and cam slots in the rotor, the latter is revolved around the rotor cylinder, being coaxial with the cylinder, and this by virtue of its inertia produces the return stroke of the actuating piston. The rotor, protected by a cover fixed to the explosion cylinder, actuates the ignition control, the coil being housed in the cover and connected to the sparking plug in the finned head of the explosion chamber.

The impact chamber provides the fuel intake, mixing and transfer chamber, and air compression chamber; it also houses the striking pin and anvil, and supports the chuck for holding the drill or bit. The frame, formed of pipes, fixed to the rotor cover and the bottom of the impact chamber, carries fuel tank, bars forming handles for supporting and guiding the tool, and an adjustable knee plate. J. F.

930. Electric Welding of Oil and Gas Lines. H. C. Price. *Oil and Gas J.*, 1929, **28** (20), 188, 302, 305.

In the 12 months ended Sept. 1, 1929, over 2,500 miles of electric-welded pipe-lines, varying from 6 to 20 inches in diameter, had been completed. The lengths of line varied from 50 to 700 miles.

All the welded pipe was equipped with ball and spigot joints, and the majority welded with a carbon content of 0·2 to 0·3.

The generators on the rolling welds use 300 amperes, and those on the tie-in welds 200 amperes, power being supplied by 6- and 4-cyl. motors respectively. The welding wire was mild steel rod.

On a long line, two or more gangs work simultaneously, each gang being divided into two units, one for the firing line, or rolling welds, and one for the bell-hole welds.

The pipe is first lined up on skids in sections of about six lengths, and the five joints welded from the top by turning the pipe on the skids. The completed sections are then connected over the ditch and the bell-hole, or tie-in, welds made by welding completely around the new stationary pipe.

After the pipe ends are throughly cleaned, the weld is applied in two beads. The first bead is applied to the upper half, the pipe turned over, and the other half welded. After the hard scale which has formed has been chipped off, the second bead is applied. This weld must be applied continuously, the pipe being turned by chain tongs.

As the pipe cannot be turned while the tie-in weld is being made, a hole must be dug for the welder below the pipe. The first weld starts from the bottom and is worked up each side to the top. The second bead is similarly applied.

The welding rod on the first bead of the firing line is $\frac{1}{8}$ in., with a current of 175 amperes. For the second bead, $\frac{5}{32}$ or $\frac{1}{4}$ in. rod, with 200–250 amperes, is used. For both beads on the tie-in weld $\frac{1}{8}$ in. wire is used.

Tensile tests on coupons from 52 welds on a 12-in. line averaged 50,980 lb.s/sq. in., practically all breaking through the weld near the junction with the bell. Hydraulic tests, using 800–1000 lbs. water pressure, showed negligible leaks.

Compared with gas-welding, the electric-welding shows a total saving in costs of 22 per cent. G. S.

931. Electric Motors in Oil Line Pumping. G. R. Prout. *Oil and Gas J.*, 1929, **28** (15), T48, T54, T56.

At the present time there is over 115,000 h.p. in electric motors in main oil pipe-line pumping stations in Texas, Oklahoma and Arkansas. The motor-driven station, whether using centrifugal or reciprocating pumps, has advan-

tages which are due to low first cost, small losses in material, less labour needed, quicker installation, electric power available for lighting, etc., altitude and water supply have less effect on location, fewer auxiliaries are required and the manifold needed is simpler. Where the centifugal pump is used, the motor-driven station will readily allow of full automatic control. G. S.

932. Report on Electric Coring. C. Schlumberger. *Petr. Times*, xxii, **561**, 720.

The name electric coring is intended to signify an operation undertaken with the object of determining, by measurements of electric resistance carried out in the uncased portion of a borehole, the nature of the rocks penetrated by this borehole. Thus the operation like ordinary mechanical coring, is directed towards obtaining geological knowledge of the strata drilled.

The resistance presented by different classes of rocks varies greatly, and forms physical parameters which can conveniently be measured in the interior of the borehole. If certain metallic ores are excepted, rocks are not conductors of electric force apart from the water inherent in them, while the solid mineral deposits are almost perfect insulators, which the current sidesteps in following the humid veins. From this the following approximation law has been deduced. The specific resistance of a rock is: (1) Inversely proportional to the volume of water inherent in a cubic metre of the rock; (2) Proportional to the resistance of this water, and therefore in an inverse ratio to the total quantity of salts dissolved in this water.

The measuring apparatus consists of three insulated cables suspended in the hole and terminating towards the bottom in three electrodes A, M and N, which are immersed in the well water. The diameters AM=r and AN=r¹ are shown greater than the diameter of the hole. The electrode A serves to send the current into the soil and the electrodes M and N to measure the difference of potential produced by ohmic effect between these two points by the passage of the current in the soil.

Then knowing the distances r and r¹, the intensity i of the current and the difference of potential ΔV between M and N it is possible to calculate δ the average resistance of the soil surrounding the measuring field AMN. If the soil is uniform the formula to calculate δ becomes

$$\delta = 4\pi \frac{\Delta V}{i} \quad \frac{r r^1}{r^1 - r}$$

which gives the required resistance. When the soil surrounding the measuring field is not uniform, calculation becomes more complicated, but yield results which are sufficiently accurate for practical purposes. H. E. T.

933. Protecting Lines Against Corrosion. G. N. Scott. *Oil and Gas J.*, 1929, **28** (17), 37, 157-158.

The critical examination of existing coatings on pipe lines with a view to defining the status of pipe line coating practice showed many instances of inefficient and wasteful methods of application, and also that the primary cause of the failure of a large part of the coatings commonly used was identified with soil action. So large in number are the variables affecting the behaviour of protective coatings, and so complicated is the interpretation of field accumulated information, that a proposal is made by the American Petroleum Institute for co-operative research between the Bureau of Standards and the manufacturers and consumers of protective coatings whereby information on the behaviour of each manufacturer's products upon actual pipe lines under field conditions may be collected and studied, since laboratory methods for determining a coating's protective value have not yet been satisfactorily correlated to the behaviour of the coating on a pipe line.

H. S. G.

934. Principles Involved in Flowing Wells. S. F. Shaw. *Oil and Gas J.*, 1929, **28** (20), 178, 299, 302.

The main factors involved in flowing wells are: (1) submergence, (2) lift, (3) diameter of flow tubing, (4) quantity of liquid, (5) back-pressure, (6) temperature of the liquid, and (7) viscosity of the liquid.

In air-lift work the pressure must be greater at the bottom of the tubing than at the top, and the greater the submergence the greater the pressure under which the gas can exist. The greater the submergence, the greater is the lifting efficiency.

The diameter of the tubing has a marked effect on gas-lift performance, and for maximum efficiency in lifting a given quantity of liquid there is a given tubing diameter.

Although back-pressures are sometimes beneficial, injurious effects are more often the result.

The raising of the liquid temperature and the consequent reduction of viscosity will lower friction losses. The heating of input gas has been found to increase the quantity of the liquid lifted.

In all lifting operations four points of production can be obtained in a tubing of given design. They are: (1) Point of no flow caused by deficiency of gas, (2) point of maximum efficiency, (3) point of maximum capacity, and (4) point of no flow caused by excess of gas.

The gas-oil ratio can be controlled by changing any one or more of the variables, *e.g.*, submergence, lift, flow tubing, quantity lifted, temperature, or discharge pressure.

G. S.

935. Operating Costs of Prime Movers (for Drilling Wells). C. L. Shirley. *Oil and Gas J.*, 1929, **28** (21), 62, 238-9.

In determining a figure for the operating cost of the prime mover information is difficult to obtain, for records are often not kept, are unreliable, may be arbitrary depending on estimation of profits, and biassed as in the case of some dealers.

Data collected from various sources show that although operating conditions vary, the cost of drilling a well may be expected to equal the average cost of previously drilled wells. This indicates that the standard of efficiency of well drilling equipment is the same in various localities, and this is in general the case in practice.

Greater part of the data used relates to rotary drilling, as this is the method most employed at present.

The prime movers of both standard cable tools and rotary tools are steam engines and boilers, either gas, oil or coal fired; internal-combustion engines gas or Diesel types; electric motors, power being supplied from individual or central stations. With standard cable tools electric installations show the lowest cost, and electrical equipment is still being improved. With rotary tools steam plant is most usual as yet because of simplicity in operation, although the cost of operating rotary tools by electric power compares favourably with averages for electric cable tools. Very meagre data are available and that is unfavourable for internal-combustion installations.

Operating costs and desirability of electric rigs over steam rigs are matters of geographical and geological location. But whilst the steam rig will remain unchanged in the future, electric rigs will be improved and operation much simplified.

J. F.

936. Electrification of Oil Fields. W. S. Toplis. *Journ. Inst. Petr. Techn.*, 1929, **15** (76), 591-610.

Electricity being recognised as the most reliable and efficient method of power distribution, the problem resolves into selection of equipment and its design to overcome troubles.

The fuel question is of prime importance, but usually residue casing-head gas is available. The design of burners and combustion chambers requires careful consideration, and the author favours the Schaefer or Hunter burner. For units of 30,000 lb. evaporation or over, induced draught and preheated air manifold should be considered.

Steam turbine generators are usually the best, but when good water is scarce large gas engines may be used. A typical engine is a horizontal twin tandem, double acting, four cycle. Diesel engine plants are more expensive than either of the other types.

Insulation is important. For the E.H.T. distribution, the ring circuit split conductor system appears to be best. The towers carrying the lines are built up of steel angles, two types being used—anchor and intermediate. The anchor towers are designed for longitudinal and transverse strains and the intermediate towers mainly for transverse strains. All the material is heavily galvanised.

The transmission line conductors are 7 No. 1/0 B. & S. hard drawn copper strand wires, the ring main conductors being solid No. 3 B. & S.

In the low tension distribution the conductors are carried on expanding steel truss poles, the lines being 2 No. 4 B. & S. hard drawn copper standard wires. The service is taken off through hand-operated switches, the lines being of 3 No. 1/0 B. & S. stranded copper wires.

Electricity was first applied to rotary drilling in the early 1920's. At first 75 to 100 h.-p. motors were tried, but it is now general to use 125 h.-p., or twin 100 h.-p. equipments.

Control gear is now receiving close attention and modern developments will bring this apparatus into the class of all magnetic contractors.

At first a 75 h.-p. single-speed motor was used for cable drilling, but a special equipment in the form of a twin motor drive has been developed.

Apart from actual drilling operations, electricity is also applied to slush pumps, inclinometers, derrick heating, pumping, dehydration, lighting, etc.

G. S.

937. Drilling on the Gold Coast, West Africa. O. A. L. Whitelaw. *Journ. Inst. Petr. Techn.*, 1929, **15** (76), 574-587.

In Appollonia, the existence of bitumen and oil was referred to in 1882. Between 1895–97 five wells to a maximum depth of 800 ft. were sunk near Bokakra. In 1923–25 two wells were sunk at Bonyero and Takinta, the maximum depth being 4005 ft. Shows of oil and gas were met with at various depths in both of these wells.

It is also reported that three borings sunk in 1910—1913 at Bokakra, Bonyero and Adul reported shows of gas and oil. G. S.

938. Some Problems in Deep Well Pumping. P. Williams. *Oil & Gas J.*, 1929, 28 (15), 37, 359.

The need for an exact knowledge of working loads is eminent, and proper alignment of the working parts of the pumping rig is essential. Counterbalance, particularly the rotative type, is by far the most flexible controlling element in proper distribution of loads throughout the equipment. In deep well pumping the counterbalancing problem is economically worthy of a specialist, as the proper adjustment of any type is an intricate physical problem.

Sucker rod loads should not exceed 22,000 lb unit stress. Tapered strings are assembled in sections. For locating the point in the rod string where it is permissible to reduce the size of rods and maintain the same unit stress in the upper portion of each section the following formula is offered:—

$$\frac{C\,(W_1 \times = W_2\,(D-X)+F)}{A_1} = \frac{C\,(W_2\,(D-X)+F)}{A_2}$$

W_1 = Weight per foot of larger rods (pounds).
W_2 = „ „ smaller rods (pounds).
D = Depth of well in feet.
F = Calculated weight of fluid (pounds); based on area of plunger times true pumping depth.
A_1 = Cross-sectional area of large rods (inches).
A_2 = Cross-sectional area of small rods (inches).
D—X = Number of feed of small rods.
C = 1·2 for polished rod upstroke speeds from 600 to 900 ins per min.
= 1·3 „ „ „ „ 900 to 1,200 „
= 1·4 „ „ „ „ 1,200 to 1,500 „
= 1.5 „ „ „ „ 1,500 to 1,800 „

C compensates for the variable inertia loads and for approximate accuracy is dependant on proper counterbalancing.

To assure proper "shouldering" of joints under operating loads the initial tension and shear in pins and the corresponding compression and shear in boxes, before application of pumping load should approximate the ultimate load which that portion of the string will carry. Many wells are operated with larger pump barrels than are necessary. W. L.

939. Flexibility in Electric Line Pumping. O. Wolf. *Oil & Gas J.* 1929, **28** (15), T–129–135.

The gradual reduction in current cost in recent years, coupled with improvements in pump design, have brought the combined efficiency of motors and suitable pumps to a point where they are superior to Diesel engine driven reciprocating pumps. The motors in general are either of the squirrel cage induction type, or the synchronous motor. The first is preferred because absence of slip rings permits its use without a fire wall, while the synchronous motor has a slightly higher efficiency and has the advantage of giving a power factor correction which may be used when the power contract calls for a charge based on a maximum demand charge expressed in K.V.A. Neither machine is suitable for variable speed, and therefore in themselves they lack the flexiblity attained with modern Diesel drive or steam pumps. The centrifugal pump has inherent qualities of regulation on account of its head-capacity characteristic, making it more suitable for motor drive than the normal type of reciprocating pump. Curves are given showing the effect of this characteristic. Unfortunately, seasonal conditions affecting viscosity of the crude react on the output of the centrifugal pump both as regards pressure and rate of flow, resulting in loss of line capacity when the viscosity is high. Various methods of partial correction, such as the use of alternative sizes of plunger, may be used with reciprocating motor driven pumps, but they are always accompanied with loss of energy. This loss may be serious in large installations. The Worthington Pump and Machinery Corporation, working to the patents of G. C. Hicks, have developed an efficient and economical means of overcoming the inherent lack of flexibility of motor-driven reciprocating pumps which depends for its action on the variation of phase relation between two plungers acting on two separate pumps driven by a common pinion shaft. Both pumps have their suction and discharge lines manifolded into one inlet

and outlet, and the plunger chambers are interconnected by means of an equalising pipe. The common pinion shaft actuates both pumps through a cut-off coupling which makes it possible to change the phase relation when desired (the pumps being stopped for this purpose), so that the discharge of the unit may be varied from zero to maximum rated capacity. The power consumption is directly proportional to the increase of line capacity, and thus capacity variation may be attained by the use of highly efficient motors without the need for speed variation. R. S.

940. Development of Flowing Well Theory. J. Versluys. *Oil & Gas J.*, 1929, **28** (20), 160, 240A–240B.

The author sets forth in detail the mathematical principles of a theory of the flow in wells. The constants in the formulæ must be ascertained by experiments, which should be performed on the basis of this theory. G. S.

Petroleum Geology (Geophysics).

941. The Mintrop Seismic Method. E. H. Neville. *Jour. Inst. Pet. Tech.* 1929, 15, 78, 569.

Modern methods of geophysics have, during the last ten years, become recognised as being of great value in giving valuable assistance to those interested in the exploitation of oil and mineral-bearing sands. The most important methods employed are gravimetric, seismic, electric, magnetic, radio-active and geothermic. Valuable information can be obtained from a geophysical examination of an area, by the application of either one or a combination of one or more of these methods. Thus ore bodies, salt domes, anticlines, synclines, and faults can be located and outlined. This paper is confined to the seismic method, which has the important advantage that it provides a means of determining, with close approximation, the depth at which underground beds are to be found. The paper gives a brief outline of the seismic method and its application as developed by Dr. L. Mintrop. The method is based on the fact that rocks with different elasticity and density transmit mechanical and acoustic waves with different velocities.

A number of small and transportable seismographs are placed along a straight line, a charge is exploded at one end of the line, and the seismic waves resulting are recorded by the instruments. Each seismograph is combined with a photographic-recording apparatus, on which the vibrations reaching the seismograph are registered, as well as the exact moment of explosion by interruption of an electric circuit. A number of such seismograph records, known as "seismograms," are obtained from each explosion. By drawing distance-time curves it is possible to show the presence of upward or downward inclinations of the rocks, faults and displacements, swallow-holes, and other irregularities in the lower formations, all of which have characteristic effects on these curves. Striking successes have attended the application of the seismic method in various parts of the world, one of the most striking of which was the accurate contouring of the surface of the Palæozoic rocks beneath the later loosed strata in Holland. H. E. T.

942. Geophysical Work in Great Britain. W. F. P. McLintock and J. Phemister. *Jour. Inst. Pet. Tech.*, 1929, 15, 76, 563.

In this article an account is given of the geophysical work which has been carried out by the Geological Survey of Great Britain. Gravitational surveys employing the Eötvos torsion balance are described, the structures investigated being a dyke of basic igneous rock intrusive into marls, a buried channel

and the associated pre-glacial topography, and a fault in carboniferous strata. Magnetic movements over a dyke of basic igneous rock are also described. H. E. T.

943. Geophysical Methods. C. Maurain. *Rev. Pétrolifère*, **323**, 745–749. June 1, 1929.

This article provides a general description of the geophysical methods employed at the present time in the study of the structure of the earth. In particular the seismic and electrical methods are outlined, these being based on the difference in density, the magnetic susceptibility, the elasticity and the electrical conductivity of various rocks. Other geophysical methods are used, however, to a limited extent, such as the study of radio-active properties, while acoustic methods have also been employed. The author points out that geophysical methods can only be applied indirectly in the search for petroliferous areas. H. E. T.

944. Geophysical Methods of Prospecting. G. A. Boutry. *Rev. Pétrolifère*. Sept. 28, 1929, **339**, 1369.

Seismic Methods : Part I.—The seismic method employed in geophysical prospecting yields results which agree very closely with those obtained by means of the torsion balance, and in Louisiana and Texas has indicated the presence of salt domes. The method has for its object the study of the geological structure of the lower strata of the earth, and this is carried out by determining the speed of propagation of sound waves through these various strata. Practically acoustic emitters are not employed as a source of sound waves, since they cannot produce sufficient initial sound energy to counteract the dissipation which occurs as the wave is propagated ; hence dynamite is universally used as the source of sound.

The fundamental physical laws on which the propagation of sound in solids and fluids depend are then discussed in a brief manner. The application of these laws to the propagation of sound waves through the earth's strata is very complicated, since they only apply to solids which are rigorously homogeneous and isotropic. As a general rule densities and Young's modulus of elasticity increase with the depth below the surface, so that it is difficult to imagine a strata which is sufficiently thick and formed of the same components which possesses similar mechanical properties in all directions. Two effects result from this discontinuity, namely : (1) As the depth increases the formation becomes more and more rigid, and as a consequence propagates sound with an increasing velocity. This rule is subject to exceptions but holds good for the majority of cases. (2) In the interior of a uniform formation the speed of sound cannot be considered uniform, since it varies with the depth and sometimes even laterally. The result is that the index of refraction of the sound waves varies, and the paths of the rays are not straight lines but curves. The only means of calculating the paths of the sound rays with the minimum amount of approximation is by assuming that the pressure of the formation is proportional to the depth, since fortunately the variation of the refractive index with the pressure is very small.

The factors which influence the speed of propagation of sound in the various formations are their physical state, humidity and state of agglomeration. According to the nature of the various rocks traversed by the sound waves, the intensity of the transmitted waves varies within very wide limits, because various formations have considerable absorbing powers. The charge of dynamite required to produce sensible records on the recording seismograph is found to be 250 grams placed at a distance of 125 kilometres. Again the frequency of the recorded vibration depends on the rocks traversed, while

the angle of emergence of the recorded waves is in most cases extremely small. This latter fact presents a difficult explanation and is in contradiction to the general laws which serve as a basis for the work of interpreting the results obtained by the seismic method. The article then concludes with an outline of the nature and causes of the natural movements of the earth. H. E. T.

945. Geophysical Methods of Prospecting. G. A. Boutry. *Rev. Pétrolifère.* Sept. 14, 1929, **336**, 1205.

Electrical Methods : Part I.—In this article the author discusses the application of electrical methods to geophysical prospecting, since by studying the propagation of either continuous or alternating currents in the earth, the presence of substances which are either particularly good conductors or very good insulators are indicated. The propagation of a direct electrical current in the earth is then discussed, and it is stated that the phenomena is characterised by the value at each point of the conductor, of the current-intensity j defined by the equation :—

$$\rho j = -\frac{\delta V}{\delta l} \quad . \quad . \quad . \quad . \quad . \quad (1)$$

where V = the potential
ρ = the resistivity of the conductor at the point under consideration.

This equation represents the general differential expression of Ohm's Law; The equipotential surfaces of the conductor and the modifications in the general theory which have to be made when the conductor is anisotropic are next discussed. The magnetic field produced by the current flowing in the conductor is then treated according to the law of Laplace, namely :—

$$dH = \int \frac{j\sigma \sin \alpha}{r^2} dl \quad . \quad . \quad . \quad . \quad (2)$$

in which dl = the length of an olemenat of a tube of force
σ = area of cross section of a tube of force
j = current density.

The value H of the magnetic field accompanying the current is obtained by integrating equation (2) throughout the volume of the conductor, but in general its value cannot be obtained. The resistivity of a conductor depends not only on its nature, but on its structure and state, and according to their structures various rocks present greatly varying resistivities. Another factor of importance is the state of aggregation of the rock, since it is known that a rock which is normally a good conductor, becomes almost a perfect insulator when it has a folded structure and the current flows at right angles to the strata.

The propagation of alternating currents of high and low frequency in the earth is now discussed, and the general theory is outlined. The existence of natural currents in the earth, and the probable causes to which they are due, are now described. These natural currents provide direct evidence for the prospector, but their existence is unfortunately limited to a very small number of cases. H. E. T.

946. Geophysical Methods of Prospecting. G. A. Boutry. *Rev. Pétrolifère.* July 27, 1929, **331**, 1027–1031.

This article describes the principles and mathematical theory of the instruments employed for research on the magnetic anomalies of the earth. A general discussion on the earth's magnetic field is first given, the properties discussed being the variation of the magnetic field and both periodic and rapid variations due to the propagation of magnetic waves having a velocity of 100 to 200 kilometres a second. The instruments commonly used in order

to determine magnetic anomalies are named the "Magnetic Variometers." Two such magnetometers are in use, one measuring the horizontal component of the magnetic field and the other the vertical component.

The mathematical theory of both these instruments is then outlined, and this is followed by a discussion on the measurement of the magnetic declination and inclination. The article concludes with a description of the earth inductor. H. E. T.

947. Geophysical Methods of Prospecting. G. A. Boutry. *Rev. Pétrolifère*, **330**, 1097–1100. Aug. 24, 1929.

In this article the author continues his discussion on the magnetic anomalies, describing the practical application of magnetometric methods. The actual construction and application of both the horizontal and vertical variometers is outlined, while short details are provided illustrating the manner in which these two instruments are set up and used during a geophysical survey.

In the execution of magnetometric measurements it is absolutely necessary to determine :—

I The temperature at which each measurement is made ;

and II A convection curve for the instrument being used, in which temperatures are plotted as abscissæ and the corresponding deviations as ordinates, the apparatus remaining in a uniform and constant magnetic field.

By this method a curve is obtained which is in general approximately rectilinear, and where gradient $a = \Delta z / \Delta t$ gives the temperature coefficient of the apparatus. H. E. T.

948. Geophysical Methods of Prospecting. G. A. Boutry. *Rev. Pétrolifère*. July 28, 1929, **330**, 1003.

In this article the author discusses the application of the Eötvos torsion balance as a means of determining the positions of salt domes, faults and anticlines. Two examples of the application of the torsion balance are given for salt domes in Texas. The discovery and mapping of salt domes, faults and anticlines is accomplished by observing the variation in the gravitational potential. H. E. T.

Coal and Shale.

949. Treatment of Crude Oils, Tars, Bituminous Residues and the Like. L. Bolgar. E.P. 319,673, Sept. 23, 1929. Appl. March 23, 1928.

This invention relates to a method of producing an artificial asphalt, and consists in mixing with crude oils, tars, residues, etc., oxidising agents such as acids or acid containing substances, for instance, acid sludge. The mixture is heated to between 120° and 200° C. and then allowed to rest until a precipitate is formed. The latter is separated from the liquid portion and heated until the acids contained in the precipitate are decomposed. The amount of acid used is from 1 to 25 per cent., calculated for concentrated acid, while the concentration is 66° Beaumé. In order to decrease the viscosity of the mixture, a suitable solvent may be added, thereby promoting the separation of the precipitate. The precipitate can be added to a further quantity of raw material and the treatment repeated without the addition of further quantities of acid. The liquid material obtained by this process may be refined, distilled or cracked, etc. W. H. T.

950. Process for Treating Carbonaceous Materials to Obtain Distillation Products such as Hydrocarbons, Metallised Coke, and the Like. Fabrique Nationale de Produits Chimiques et d'Explosifs S. A. E.P. 296,443, Oct. 24, 1929. Appl. Aug. 29, 1928. Conv. (Bel.), Sept. 2, 1927.

This comprises the addition to the carbonaceous materials of mineral oxidising agents, such as iron oxide, manganese oxide, or alkaline or alkaline earth nitrates or carbonates, to the extent of about 15 per cent. of the material treated. Mixtures thus obtained are then subjected to carbonisation. The volatile products of distillation are condensed to obtain a tar similar to crude petroleum. The solid residue is coherent and by a suitable selection of the additions may be used as a metallurgical coke. W. L.

951. Production of Hydrocarbons of Low Boiling Point from Oils or Solid Fuels. R. E. Goldsbrough and H. Tevis. B.P. 320,619, Oct. 14, 1929. Appl. April 15, 1929.

Highly-heated oil or pulverised solid fuel, together with superheated steam, is injected into a chamber lined with the catalyst. Here the mixture is gasified, and subsequently passed through a conduit also lined with the catalyst, where the temperature is reduced gradually to below the gasifying point. Vapours are cooled and condensed out of contact with the catalyst. The catalyst consists of 14 pts. silica, 9 pts. sodium silicate, 4 pts. alumina, 2 pts. zinc oxide, a trace of magnesite and a trace of lime. W. S. C.

952. Process for the Purification, Hydrogenation and Desulphurisation of Liquid or Gaseous Hydrocarbons as, for example, Oils or other Liquid or Gaseous Products of Carbonisation and the like. Heliodor Rostin. E.P. 286,255, Sept. 2, 1929. Appl. March 1, 1928.

This invention relates to the purification, hydrogenation and desulphurisation of liquid or gaseous hydrocarbons, such as those produced during the carbonisation of fuel. The process is carried out by passing the hydrocarbons to be purified along with sulphuretted hydrogen over finely-divided iron maintained at a temperature of 200–300° C. Finely-divided iron is particularly suitable for use, since it converts unsaturated hydrocarbons into saturated hydrocarbons quite readily. The hydrogen sulphide is decomposed by the reduced iron, so that the sulphur combines with the iron while the nascent hydrogen liberated reacts with the substance under treatment. Crude benzole, a brown, disagreeable smelling liquid, which suffered a loss of 20 per cent. after shaking with sulphuric acid, was obtained after treatment by this process as a water white liquid with an absolutely pure smell. This product had a loss of less than 1 per cent. on shaking with sulphuric acid. The best form of iron to use for the purpose of this invention is that obtained from the ore "Minette," because (*a*) it can be obtained in granules of the exact predetermined size, and (*b*) because the iron is finely distributed in the porous structure, thus offering a large effective reaction surface. H. E. T.

953. Production of Low B. P. Oils and other Products by Destructive Hydrogenation of Coals, Oils, etc. I. G. Farbenind. E.P. 287,855, Sept. 26, 1929. Appl. March 26, 1928. Conv. (Germany), March 26, 1927.

In the high-pressure catalytic destructive hydrogenation of coal, tars, oils, etc., control of reaction temperature is of prime importance. The present invention provides for temperature control partly in a direct manner by means of the substances, particularly the hydrogenating gas, admitted to the reaction chamber; and partly in an indirect manner by means of substances, part or all of which take part in and/or result from the reaction. The indirect

heating is effected by passing the hot substance in a spiral path through one or more annular chambers between the reaction chamber and the outer pressure wall. Thus the hydrogenating gas, preheated to 400° C., may be passed around the contact chamber, then further heated electrically and passed into contact with the coal or oil, etc., which has been preheated to a temperature approaching that of the reaction. If desired, heat exchange may be effected by passing the products in a spiral path through an outer annular chamber, or excess of hydrogenating gas may be passed through this chamber, only a portion being passed through the inner annular chamber to the reaction.

In this way the reaction temperature is maintained constant in the greater part of the reaction vessel, fluctuations in temperature are counterbalanced, and injurious radiation losses are prevented. L. H. A.

954. Method of and Apparatus for Carbonising or Distilling Material. International Combustion, Ltd., and S. McEwen. E.P. 316,734, Aug. 8, 1929. Appl. June 8, 1928.

The patent describes a retort adapted for carbonisation or distillation of material constructed as described in E.P. 316,901. The construction is such that the products may be withdrawn either through the perforations in the endless band or through the articulated chain grate or from the bottom of the retort as desired. W. H. T.

955. Hydrogenation of Grosny Mazout. W. Ipatiew, N. Orlow and M. Bielopolsky. *Brennstoff-Chemie*, Sept. 1, 1929, 346-347.

The sample of the residue investigated had the following constants; Sp. gr., 0·875; flash point, 126° C.; viscosity, 36; solidifying point, 35° C.; paraffin content, 10 per cent.; pitch content, 28 per cent.; distilling up to 300°,—2·5 per cent.

Cracking was conducted in a rotatable nickel chromium retort provided with a valve, manometer and thermometer, and which was heated in an electric furnace. During the heating of the autoclave the pressure gradually increased up to a point, then slowly declined, and in all cases the pressure at the end of the experiment was below the starting pressure. The spirit obtained was water white in colour, but possessed the odour of amines. When treated with sulphuric acid there is little action. The kerosine fraction darkens quickly in air, but when treated with 5 per cent. of 1·84 sulphuric acid and soda, then distilled, 76 per cent. distils as a colourless stable oil. The residue, after distillation of the spirit and kerosine, is a heavy oil which on further hydrogenation behaves like the original residue.

Catalysts, such as oxide of iron, aluminium, or copper, appear to have little effect on the process.

The spirit obtained is of low density, boiling up to 35° C.—14 per cent., up to 95° C.—54 per cent. and up to 150° C.—96 per cent.

The kerosine fraction distils between 150 and 300° C. and has a gravity of about 0.820. The best results appear to yield 34 per cent. spirit and 30 per cent. kerosine. R. P.

956. Production of Viscous Oils from Brown Coal Tars or their Distillation Products. J. Y. Johnson, Assr. to I. G. Farbenind. Akt. E.P. 316,701, Aug. 8, 1929. Appl. May 11, 1928.

The patent describes a process for the production of viscous oils from brown-coal tar or such fractions of the same as are substantially free from benzine or paraffin wax. They may also have been subjected to a preliminary treatment such as hydrogenation, oxidation, dehydrogenation, etc.

The initial materials are exposed at between 100° and 200° C. to the action of metal chlorides which are capable of facilitating the introduction of alkyl groups into aromatic nuclei after the manner of Friedel-Crafts reaction. By this means the formation of low-boiling constituents is completely or almost completely prevented. W. H. T.

957. Process and Apparatus for Treating Solid and especially Bituminous Substances. J. Y. Johnson, Assr. to I. G. Farbenind. Akt. E.P. 320,147, Oct. 10, 1929. Appl. July 23, 1928.

The patent describes a process and apparatus for the heat treatment of solid materials, in particular the low temperature carbonisation of bituminous fuels. The raw material is allowed to slide down through heated pipe retorts mounted at such an angle that the charge travels automatically. Means are provided for preventing the charge from completely filling the retorts, thus leaving a free space above the charge through which the gaseous products of carbonisation may be drawn. Baffles are provided in the spaces connecting the retorts in order to regulate the depth of the charge. The material to be treated is introduced by means of a hopper in an amount corresponding to and automatically controlled by the amount of fuel drawn off at the outlet. The charge is heated indirectly by means of hot combustion gases passed into circulation by means of nozzles arranged in a mixing chamber of the injector type. W. H. T.

958. Low Temperature Carbonisation. The Pehrson Process. *Petr. Times*, 1929, **22**, 702. (Abstract of paper read by Dr. W. R. Chapman to the Society of Chemical Industry.)

In the absence of profit and loss accounts over a period of several years' working the following conditions should be satisfied if a low temperature carbonising plant is to prove a commercial success. High efficiency of heat transfer to the coal without overheating, uniformity of products, production of semi-coke in lumps strong enough to bear transport and handling without undue disintegration, production of gas of high calorific value, maximum production of light oils with a minimum of tarry constituents, and actual low temperature of operation. The author considers that the Pehrson process conforms to all these conditions.

The plant consists of two internally heated rotary drums, both carried on the same axis. The first drum acts as a drier and preheater for the coal charge, the preheated material being made to pass continuously to the second or carbonising drum, without appreciable mixture of the respective gases. The carbonising medium consists of a portion of either the gases or vapours from the carbonising drum, which are reheated in a recuperator either after or before condensation of oil vapours. Around the periphery of each drum are louvre-like openings, so arranged that the charge cannot fall into the gas passages; the hot gas at about 550–600° C. is admitted only to those louvres on which the charge rests, whereby the highest possible efficiency of heat transfer is obtained. The louvres carry the charge well up the side of the drum, when it falls back without sliding or abrasion. The recuperator may be heated by producer gas; the flue gases are utilised in the preheating drum, from which they are drawn off in two fractions: the first at 100° C. contains most of the water, and is wasted; the second at higher temperature is recirculated.

One ton of coal slack yields on an average:

Gas (950-1000 B.Th.U./c. ft.)	2750 c. ft.
Oil (total)	20 gal. approx.
Semi-coke (14 per cent. volatiles)	15 cwt.

The oil contains no suspended carbon; about 44 per cent. distils over up to 250° C., and from 76–83 per cent. up to 360° C.; the pitch averages about 20 per cent. The semi-coke ignites readily, and burns more pleasantly than other semi-cokes, giving a smokeless flickering flame. By varying the conditions coking slacks can be converted into lumps of semi-coke of almost any desired size. L. H. A.

959. Separation of the Constituents of Low Temperature Tar. C. F. Morgan and D. D. Pratt. E.P. 318,116, Aug. 28, 1929. Appl. Apr. 28, 1928.

In specification No. 307, 382 a process for treating low-temperature tar and its distillates is described. The fraction hitherto regarded as the phenol fraction of approximately 25–30 per cent. can be separated into a fraction 10–15 per cent. of compounds soluble in alkali, the residue being asphaltic or resinous substances insoluble in aqueous alkali but apparently soluble in alkali phenolates.

The phenolic bodies constituting the 10–15 per cent. fraction are treated with low boiling petroleum whereby certain phenols are dissolved and isolated by distilling the petroleum, when they remain in the form of a crystallisable oil, boiling between 180° and 200° C. The insoluble residue is a viscous black mass which is an efficient binder for coal dust. After heating to 100° for some time the fusibility of the residue is decreased, and it sets to a soft pitch.

If the residue is treated with ether or benzene it may be separated into two products, resinols C and resinols D. The latter pass into solution, from which they may be precipitated as an amorphous yellow or brown solid by petroleum ether. The solution separated from this solid may be evaporated to obtain a viscous black oil.

Resinols C and D may be used as a basis for lacquers or varnishes by dissolving in methylated spirit and may be applied to wood in the same manner as French polish.

Further the resinols C and D may be condensed with formaldehyde to give synthetic resins for use in varnishes or lacquers, and any of the products described may be condensed with formaldehyde in presence of acid or alkaline catalysts to form resins for use in moulding compositions. R. P.

960. Recovery of Gas Tars from their Emulsions with Water. R. R. Ripley and S. C. Schwarz. U.S.P. 1,274,222, Aug. 13, 1929. Appl. April 22, 1924.

The object of this invention is to recover tar from the viscous hydrocarbon-water emulsions which are by-products in the manufacture of town gas from crude petroleum, fuel or gas oil. The process is adapted to deal with oil-gas and water-gas tar emulsions which contain a high percentage of solid matter and a water content of between 30 and 85 per cent.

A relatively small proportion of a demulsifying agent is added to the emulsion and the mixture is heated rapidly to boiling point at atmospheric pressure, in order to reduce viscosity and promote intimate mixing. The heating is then continued to a temperature not exceeding 320° F. under increasing pressure whereby vaporisation is avoided. When coalescence under these conditions has taken place, heating is discontinued and the pressure allowed to return to atmospheric. The mixture is finally separated into its components after a suitable period of quiescence. W. H. T.

961. Conversion or Purification of Hydrocarbon Fluids. H. Rostin. U.S.P. 1,732,111, Oct. 15, 1929. Appl. Feb. 29, 1929.

The patent describes a process of hydrogenating carbon containing compounds by conducting them together with hydrogen sulphide over finely

divided iron in the form of reduced ore at a temperature of approximately 200° C. This temperature is stated to be sufficiently high to cause the release of nascent hydrogen and its reaction with the carbon containing compounds.

W. H. T.

962. Distillation of Coal, Lignite or the Like having High Contents of Volatiles. Société de Rechercheset de perfectionnements Industriels. E.P. 294,568, Oct. 18, 1929. Appl. June 18, 1928. Conv. (Gr.) July 26, 1927.

This consists of a process of rapid distillation, in superheated steam, at about 500°c., of agglomerates of finely divided coal, lignite or the like containing about 26 to 35 per cent. of volatile matter. The fuel is agglomerated by means of a binding material. A hydrocarbon binding material is employed which, when incorporated in the agglomerate and distilled at 500° C. forms substantially no pitch, does not distil off completely, but undergoes some decomposition, and produces a substantial amount of coke. Anthracene oil is a suitable material. The distillation takes about four hours to complete, and the fuel obtained is of a very compact nature and has substantially the properties of anthracite. This residue may be redistilled at 700° C. to 1000° C. with the production of a residue having the properties of metallurgical coke.

W. L.

963. Treatment of Oils, Tars, or Pitches, from Coal to Modify their Viscosities at Predetermined Temperatures. South Met. Gas. Co., H. Pickard and H. Stanier. E.P. 316,897, Aug. 6, 1929. Appl. March 3, 1928.

A difficulty that has been met with in the use of coal tar for road-making and like purposes is that the tar may become unduly soft when exposed to the heat of the sun, and unduly hard in cold weather. It is stated that certain naturally occurring carbonaceous materials, such as peat and coal, can be incorporated with oils, tars or pitches, with a high degree of dispersion, with the result that a relatively small proportion has a considerable effect on the temperature-mobility of the tar. Moreover, the latter acquires certain special characteristics, such as a high degree of elasticity, not imparted by the known fillers. Coal or analogous bituminous material is dispersed in coal tar at a temperature of about 300° C. in proportion, not exceeding 15 per cent. of the final dispersion. The latter has a Hutchinson consistency lower than 100 seconds at temperatures of 122° F. and upwards.

W. H. T.

964. Treating Carbonaceous Material for Fuel. Trent Process Corporation, Assr. to W. E. Trent. E.P. 293,454, Sept. 25, 1929. Appl. June 25, 1928.

A process is described of treating finely divided carbonaceous material according to the processes described in E.P. 151,236 and E.P. 183,430. It consists of the following steps carried out separately: first controlling the liquid dilution and temperature of the materials during the agitation treatment so as to cause the oil and fine carbonaceous particles to form agglomerates of desired predetermined sizes capable of being carried by the moving liquid, and second causing the agglomerates to coalesce into larger masses while continuing the agitation. The agglomerates resulting from the first step are separated from the water in which they are contained and then agitated in the second step in a clean body of water. W. H. T.

ABSTRACTS

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